ASD-TDR-62-230

# DETERMINATION OF ENGINEERING PROPERTIES OF MAR-STRAINED STEELS

TECHNICAL DOCUMENTARY REPORT NO. ASD-TDR-62-230

August 1962



SAPPHIRE ANNIVERSARY

45

YEARS OF MATERIALS PROGRESS

Directorate of Materials and Processes
Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio

Project No. 7381, Task No. 738103

(Prepared under Contract No. AF 33(616)-7655 by General Electric Company, Evendale, Ohio; R. E. Yount, Author)

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6-157C, 800, 10-8-62

# FOREWORD

This report was prepared by the Materials and Processes Sub-Operation of the General Electric Company, Evendale, Ohio under USAF Contract No AF 33(616)-7655. The contract was initiated under Project No. 7381, "Materials Application," Task No. 738103, "Data Collection and Correlation." The work was administered under the direction of Directorate of Materials and Processes, Deputy Commander/Technology, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio. Mr. V. F. Lardenoit was the project engineer.

This report covers work done from 15 November 1960 to 12 January 1962.

The studies were conducted by the following General Electric personnel: R. E. Yount, project engineer and T. K. Redden, project manager.

In addition, J. D. Marble contributed greatly to planning and analysis of the engineer property evaluations and Dr. W. H. Chang gave helpful guidance in the studies of Mar-Strain response. D. J. Kroeger, W. Cress and many other Material and Processes personnel contributed to the work. Mrs. Dorothy Hammond prepared the manuscript.

AD289609


## ABSTRACT

The Mar-Strain response (the addition of strain and aging to quenched and tempered steels) was determined for eight alloys representing four classes of steel: low alloy martensitics, secondary hardening martensitics (hot work die steels), semi-austenitic and martensitic stainless. All four alloy classes responded to the process by demonstrating yield strength increases of 10 - 20%. It was found that the tempered structure and strain hardening characteristics were the most significant factors controlling the Mar-Strain response.

Two alloys were selected for determination of their engineering properties including uniaxial tensile, fatigue and center notch properties and biaxial performance in sub-scale cylinder tests. One alloy, Ladish D6AC (.46%C, 1%Cr, 1%Mo), was capable of being Mar-Strained to a 275,000 psi .2% yield strength. A second alloy, Modified S-5 (.48%C, 2%Si, .5%Mo, .25%V), was capable of being Mar-Strained to a 300,000 psi .2% yield strength. Mar-Straining was found to increase the fatigue strength of both alloys. The process was adequately demonstrated in the sub-scale pressure vessel tests. Burst strengths (greater than 350,000 psi hoop) achieved with the Mar-Strained S-5 cylinders were higher than any previously reported for a homogeneous material.


This report has been reviewed and is approved W. P. Conrardy

W. P. CONRARDY
Chief, Materials Engineering Branch
Applications Laboratory
Directorate of Materials & Processes

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# I. INTRODUCTION

The emergence of rocketry as a key area in this country's defense efforts has been accompanied by an assault on the materials problems associated with rocket systems. One of the larger such activities has been the development of better materials for high strength, light weight pressure vessels, such as cases for solid propellant rocket motors. On a strength/density ratio basis it has been shown that certain types of steels have attractive combinations of strength and ductility for such applications and that these properties can be enhanced by special processing techniques.

Early in 1960, the Flight Propulsion Laboratory Department of General Electric Company began an investigation of a previously unexploited technique for improving the properties of steels. This technique involved imposing small amounts of plastic strain to a quenched and tempered steel, followed by re-tempering or aging. This process, which was named "Mar-Straining", was found to produce appreciable increases in yield strength without significant losses of ductility. It appeared to be especially well suited for application to symmetrical components, such as pressure vessels, where uniform plastic strain can be achieved by hydrostatic pressurization. It was believed use of the process would not require expensive capital equipment, nor involve long and costly development of practical manufacturing techniques.

The early investigation of this process yielded highly encouraging results which were obtained not only with laboratory specimens but also with six inch and forty four inch diameter cylinders. When these results were presented to the Load-Bearing Materials Section of the Application Laboratory

Manuscript released by the author January 1962 for publication as an ASD Technical Documentary Report.

of ASD, the high potential of the process was recognized; and it was realized that for the process to be useful to designers it was necessary that detailed information be gathered concerning the engineering properties of Mar-Strained steels. It was further necessary to determine more specifically what steels or classes of steels are most amenable to improvement by the process.

The present contract was negotiated in order to ascertain the properties obtainable by Mar-Straining four classes of steels:

1) low-alloy martensitic
2) secondary hardening martensitic (hot work die steels)
3) semi-austenitic stainless
4) martensitic stainless

Additionally, it was planned to determine detailed uniaxial and biaxial engineering properties on two steels selected from those which appeared to be most benefited by Mar-Straining. This report presents the information and data obtained as a result of the work accomplished.

## II. OBJECTIVES

The specific technical objectives were to be met in two phases of effort:

### Phase I Objective

Determine the Mar-Strain response of four classes of steels.

a) Low alloy martensitic

b) Secondary hardening martensitic

c) Semi-austenitic stainless

d) Martensitic stainless

### Phase II Objectives

On the basis of their Mar-Strain response, select two steels for further study to determine engineering property data, both uniaxial and biaxial, in the Mar-Strained condition. The steels selected were to be representative of two different strength capabilities, 275,000 and 300,000 psi, .2% yield strength.

The uniaxial properties were to include tensile, notch and fatigue using conventional test specimens. The biaxial properties were to be obtained using 6 inch diameter cylinders with the stress, both for Mar-Straining and for testing, applied by internal hydrostatic pressure. Properties obtained were to include circumferential (hoop) yield strength, burst strength and low cycle fatigue resistance.

## III. SUMMARY AND CONCLUSIONS

The Mar-Strain response of four steel alloy classes, low alloy, secondary hardening, semi-austenitic stainless and martensitic stainless, was determined using small amounts of pre-strain and short aging times. Several conclusions were obtained.

1) Alloys which have been tempered to produce precipitated metastable compounds of carbon or nitrogen (e.g., epsilon carbide) require aging in the temperature range of 300 to 400°F.

2) Alloys which have been tempered so as to have precipitated more stable and/or complex compounds of carbon (e.g. $Fe_3C$) and nitrogen ($Fe_4N$) must be aged in the temperature range of from 600 to 800°F to achieve optimum aging response.

3) Ductility, as measured by percent reduction in area and elongation was not appreciably affected by pre-strain of .4% or less.

4) The magnitude of the yield strength increase obtainable by Mar-Straining any alloy was dependent on the strain hardening characteristics of the alloy, the larger the strain hardening exponent the larger the increase in strength.

The engineering properties of two alloys were investigated. One alloy, Ladish D6AC, was capable of Mar-Straining to a 275,000 psi yield strength. A second alloy, Modified S-5, was capable of 300,000 psi yield strength. Properties investigated were tensile, fatigue, internal notch and biaxial properties. Several important conclusions were obtained.

1) Although the aging response and strain hardening characteristics affect the magnitude of the strength increase available from Mar-Straining, the major amount of spread in tensile properties was caused by the inherent spread in the heat treated properties of the alloy.

2) The notch toughness of the alloys as measured by notch strength and $K_c$ values, was decreased slightly by Mar-Straining. This decrease in toughness is only significant to large defects introduced into the Mar-Strained alloy after pre-straining and aging. Data in the literature indicate that the Mar-Strain process would be expected to be beneficial in increasing the notch toughness as related to sub-critical defects present in the alloy before straining and aging.

3) Under tension-tension loading, Mar-Straining produced approximately a 20% increase in fatigue run-out stress over the heat treated condition for both alloys.

4) Mar-Straining was successfully applied to sub-scale pressure vessel hardware. Three cylinders of each alloy were successfully pre-strained and aged. Burst strengths $\left(\frac{PD}{2T}\right)/1.13$ of five of these cylinders were equivalent to the tensile ultimate strengths of the alloys.

5) Burst strengths of the Modified S-5 alloy were higher than any which have been previously reported for a homogeneous material. The hoop strengths at burst were 354,000, 360,000, and 362,000 psi with full-shear fracture in all three cylinders. The 362 ksi burst was achieved after cycling to 312 ksi nine times before the final burst test.

## IV. RECOMMENDATIONS

The recently completed efforts have suggested several other areas which need investigation before the full potential of the Mar-Strain process can be realized.

1. Certain applications which are now utilizing alloys of the four classes of steels investigated require knowledge of the elevated temperature properties of these alloys. In order to determine usefulness of these alloys in the Mar-Strained condition for these applications, the high temperature properties should be investigated.
2. Further effort should be expended in the application of the process to sub-scale hardware to further substantiate the reliability of Mar-Strained components. Data obtained indicate that if the pre-strain operation has been successfully accomplished, failures below the pre-strain stress should not occur. The application of the process to full scale hardware is also indicated by the results obtained.
3. Although not essential to the study of the effects of Mar-Strain process on engineering properties, further studies should be made to determine the exact metallurgical mechanism involved in the process.
4. Further investigation of the notch properties should be made. Such variables as thickness, notch severity ($K_t$-stress concentration) and types of loading (low and fast strain rate, impact, etc.) should be studied. Attempts should also be made to clarify the effect of Mar-Straining on specimens containing notches to

determine if the suspected increase in notch toughness, because of notch blunting or addition of compressive stresses, is present.

5. In the fatigue area the effect of Mar-Straining on properties under various types of loading should be more fully investigated.

6. Because of its potentially high strength with good notch toughness, the Modified S-5 alloy should be more fully investigated in the heat treated and Mar-Strained condition.

# V. PHASE I

## MAR-STRAIN RESPONSE

### A.) Background

In the past few years, several investigations have shown that a small amount of plastic strain imparted to certain steels, previously quenched and tempered, followed by a re-temper or aging treatment, can significantly raise the yield point. Stephenson et al [1] postulated several metallurgical phenomena which could be responsible for this strengthening effect, including:

1. Cottrell pinning
2. Retained austenite
3. Substructure formation

The data which they originally obtained did not conclusively demonstrate the nature of the prime strengthening mechanism, but did suggest that a substructure induced by pre-strain combined with the diffusion of carbon atoms to that substructure could be responsible. Stephenson and Cohen [2] have recently reported metallographic evidence that the carbide dispersion which occurred on quenching and tempering had been altered by the strain and post aging treatment. This change in carbide dispersion can be caused by precipitation of carbides during aging which were not precipitated during the original heat treatment (a decrease in the solubility of the carbides at the tempering temperature) or could be brought about by solutioning of the originally precipitated carbides with subsequent migration to the strained areas in the structure and precipitation at these sites. In either event, carbides locate themselves at dislocation sites introduced during the pre-straining and discourage further movement of these dislocations under stress until the pre-straining stress has been surpassed. How much the pre-straining

stress is surpassed depends on the effectiveness of the aging conditions.

The strength obtained by Mar-Straining is also affected by the strain hardening characteristics of the alloy. Rapid strain hardening response allows large increases in yield strength with small amounts of pre-strain. This characteristic increases the dislocation density and contributes to the effectiveness of the aging mechanism. Strain hardening characteristics are controlled by alloy composition and heat treatment and are dependent on the resultant microstructure.

All of the alloy classes included in this study achieve their maximum strength through an appropriate solutioning or austenitizing temperature to promote the formation of martensite upon quenching to or near room temperature. The martensite formed is then tempered to achieve the desired strength level by controlling the precipitation of metastable or stable carbides of iron or of complex carbides or compounds of other alloying elements. Carbide dispersion is controlled for either high strength (also good strain hardening) with adequate ductility, or to give lower strength with greater ductility.

The one generally accepted mode for the sequential decomposition of martensite with increased tempering temperature is:

1) Precipitation of a metastable carbide (epsilon carbide, $Fe_{2.4}C$) from the high carbon martensite leaving a lower carbon martensite of approximately .25% carbon. This stage is present up to approximately $600^{\circ}F$ for an alloy with less than 1% silicon and up to $700-800^{\circ}F$ for alloys with 1% or more silicon.

2) These two products, epsilon carbide and low carbon martensite, then decompose to ferrite (alpha iron) and cementite ($Fe_3C$); this decomposition begins at the end of the first stage at 600 or $800^{\circ}F$ depending on silicon content.

3) Following closely behind or in conjunction with and at the expense of the formation of cementite, complex carbides and compounds of other alloying elements are precipitated.

4) Following precipitation, with increased temperature and time, agglomeration of these complex compounds and cementite occurs.

Two areas of this decomposition are used to achieve the high strength available in steels. Control of epsilon carbide dispersion is used to obtain maximum strength for steels tempered at low temperatures (as 400-600°F in the low alloy martensitics). For steels tempered at higher temperatures (800 - 1050°F), strength is achieved by control of the precipitation and dispersion of the more stable complex compounds (carbides, nitrides, etc.).

Therefore, the tempered structure subjected to the Mar-Strained process has a strong influence on the strength which can be realized. The structure affects the strain hardening, hence influences the amount of pre-strain which is required to achieve a desired yield strength. It also affects the aging reaction since the compounds precipitated will vary in stability and solubility. For example, stable compounds formed at high tempering temperatures will require higher aging temperature to promote reformation of the compounds at the dislocations added by pre-straining.

Prior to negotiation and initiation of this contract, the information and data which had been obtained on alloys subjected to the Mar-Strain process pertained to only one alloy steel class, low alloy martensitic. Alloys investigated to determine their response to Mar-Straining in this initial study were:

1. Hy-tuf - Heat treatable to 200,000 psi, .2% yield

2. 300M - Heat treatable to 240,000 psi, .2% yield

3. WCM-4 - Heat treatable to 250,000 psi, .2% yield

4. WCM-1 - Heat treatable to 275,000 psi, .2% yield

(See Table 1 for chemical composition)

The choice of these alloys was based on prior knowledge of their strain hardening characteristics. The microstructure of these alloys, heat treated to their maximum strength level (approximately 600°F), consisted of essentially the same microconstituents (epsilon carbide and low carbon martensite) and varied only in carbon content.

All of these alloys were investigated (with the exception of Hy-Tuf) in the 400 and 600°F tempered condition. In these preliminary experiments, pre-strains up to 1% and aging temperatures 50°F less than the original tempering temperature were used. Aging time was four hours. Sheet thickness was .080 to .100 inches.

The data obtained are shown in Figures 1 through 8. These results prompted the following general comments:

1. The greater part of the increase in .2% yield strength was achieved with as little as .4% pre-strain.

2. The ultimate strength of these alloys, as Mar-Strained, remained nearly unaffected up to approximately .4 to .5% pre-strain. At this point, when bars containing more than this amount of strain were tested, the new yield point and ultimate strength were equal and were increased concurrently with the amount of pre-strain.

3. Although the as heat treated yield strength of these alloys tempered at 400°F was lower than that obtained by tempering at 600°F, nearly equivalent yield strengths were obtained after Mar-Straining 0.4%.

## TABLE I

## ALLOY COMPOSITIONS

| Alloy | C | Mn | Si | Ni | Cr | V | Mo | W | Other |
|---|---|---|---|---|---|---|---|---|---|
| 300M | 0.40 | 0.75 | 1.6 | 1.8 | 0.9 | 0.1 | 0.40 | - | - |
| D6AC | 0.46 | 0.75 | 0.2 | 0.6 | 1.0 | 0.1 | 1.0 | - | - |
| WCM-1 | 0.50 | 0.80 | 2.0 | 5.0 | 1.0 | 0.1 | 0.5 | - | - |
| WCM-4 | 0.40 | 0.80 | 2.0 | 5.0 | 1.0 | 0.1 | 0.5 | - | - |
| WHC | 0.50 | 0.50 | 1.5 | 4.0 | 1.0 | 0.25 | 0.5 | 1.0 | 3.0-Co |
| Mod. S-5 | 0.50 | 0.80 | 1.8 | - | - | 0.25 | 0.5 | - | - |
| HyTuf | 0.25 | 1.3 | 1.5 | 1.8 | - | - | 0.4 | - | - |
| Vascojet 1000 | 0.40 | 0.5 | - | - | 5.0 | 0.5 | 1.3 | - | - |
| AM-355 | 0.13 | 0.95 | 0.25 | 4.3 | 15.5 | - | 2.75 | - | 0.10-$N_2$ |
| 422 | 0.20 | 0.75 | 0.25 | - | 12.0 | 0.25 | 1.0 | 1.0 | - |

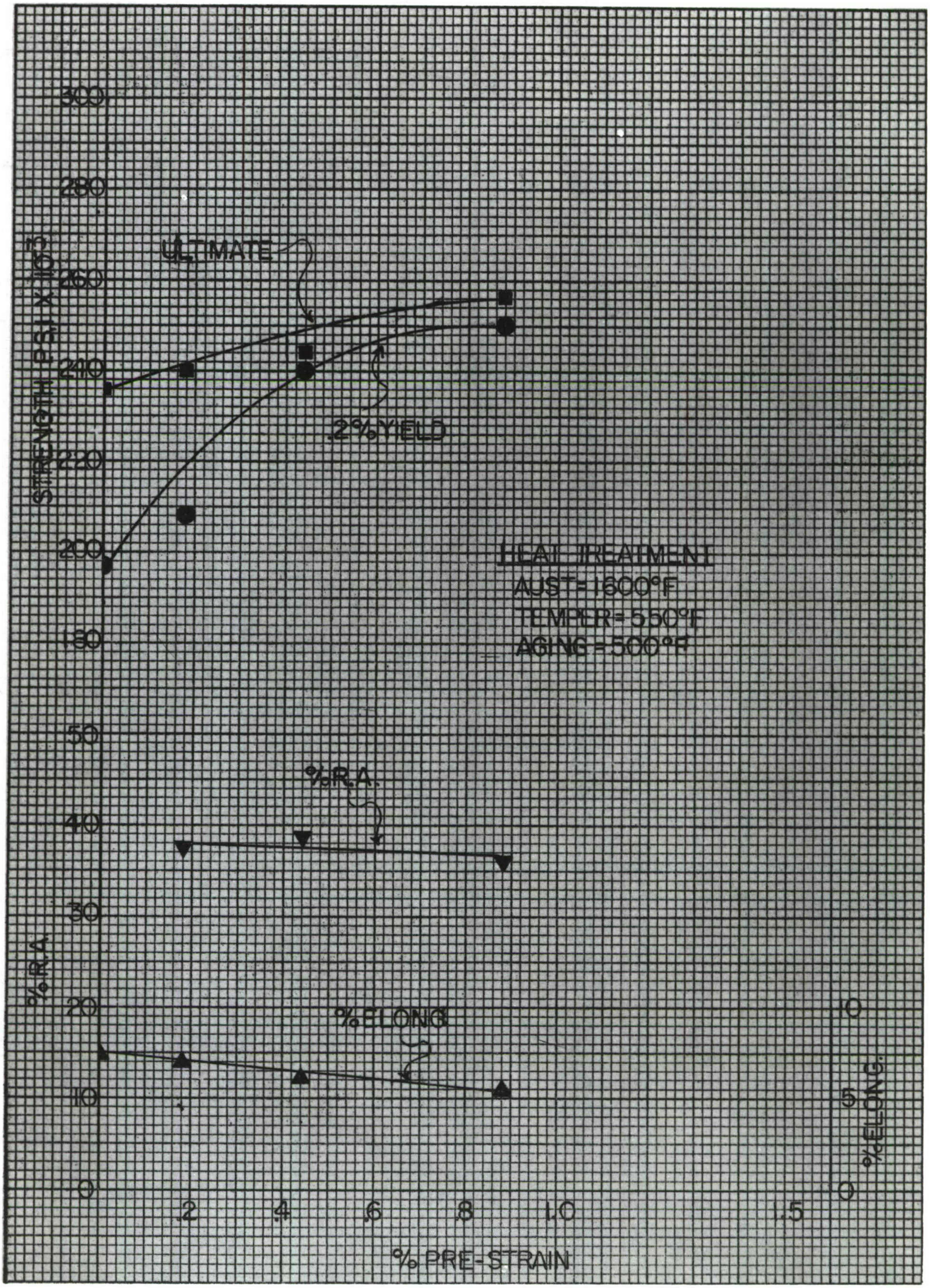



FIGURE I

TENSILE PROPERTIES OF MAR-STRAINED HY-TUF

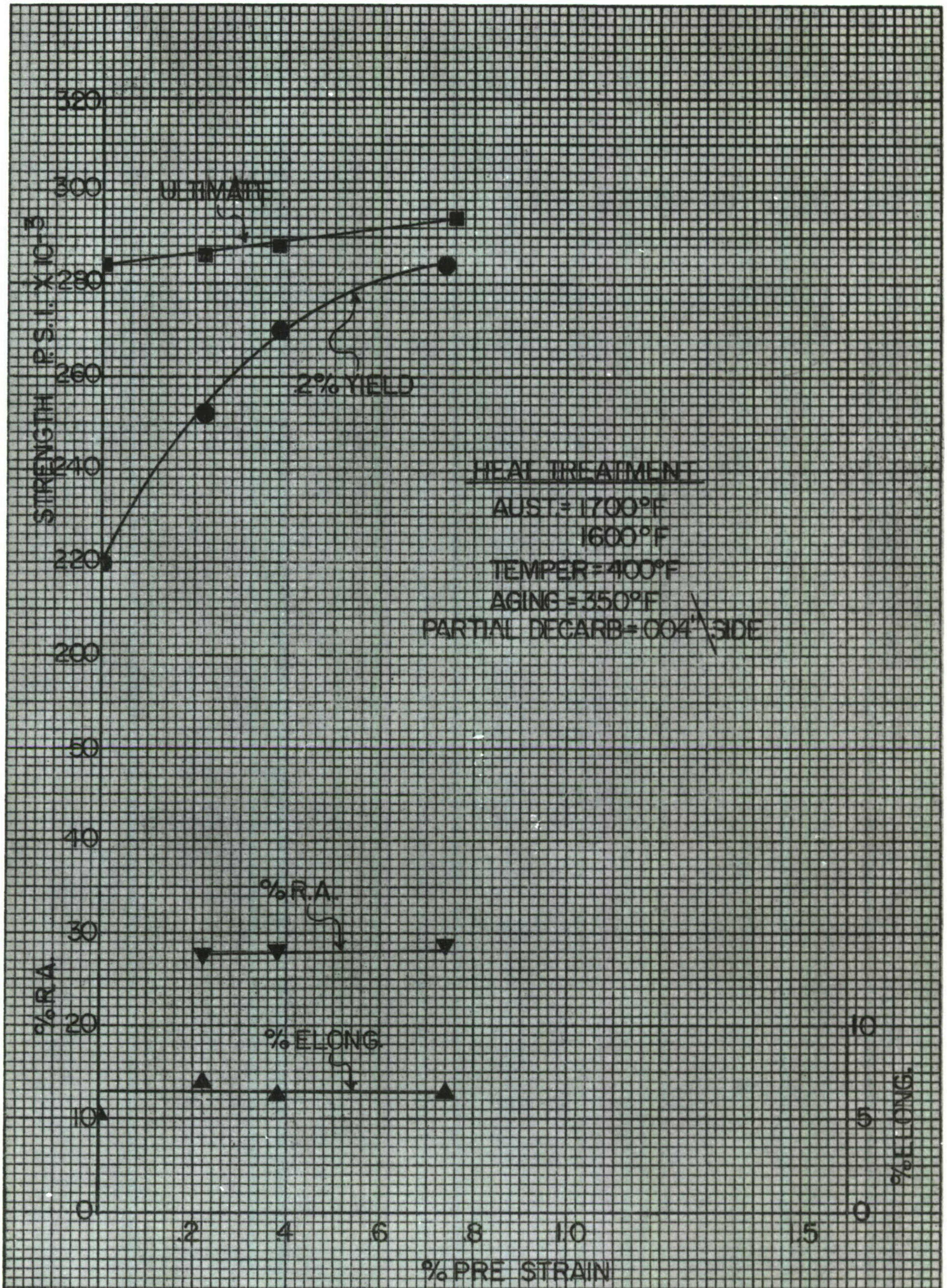



FIGURE 2
TENSILE PROPERTIES OF MAR STRAINED
300-M

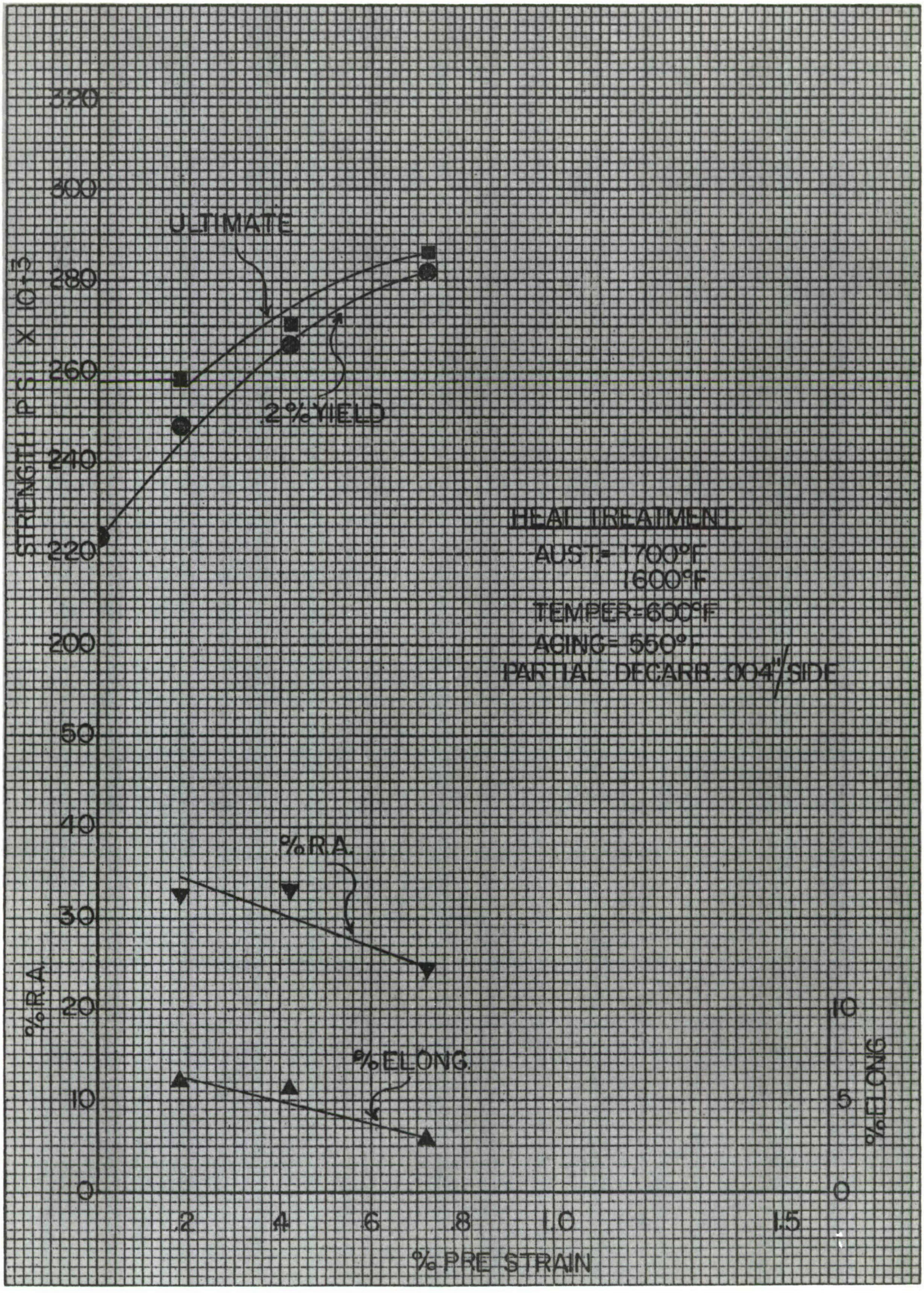



FIGURE 3
TENSILE PROPERTIES OF MAR-STRAINED
300M

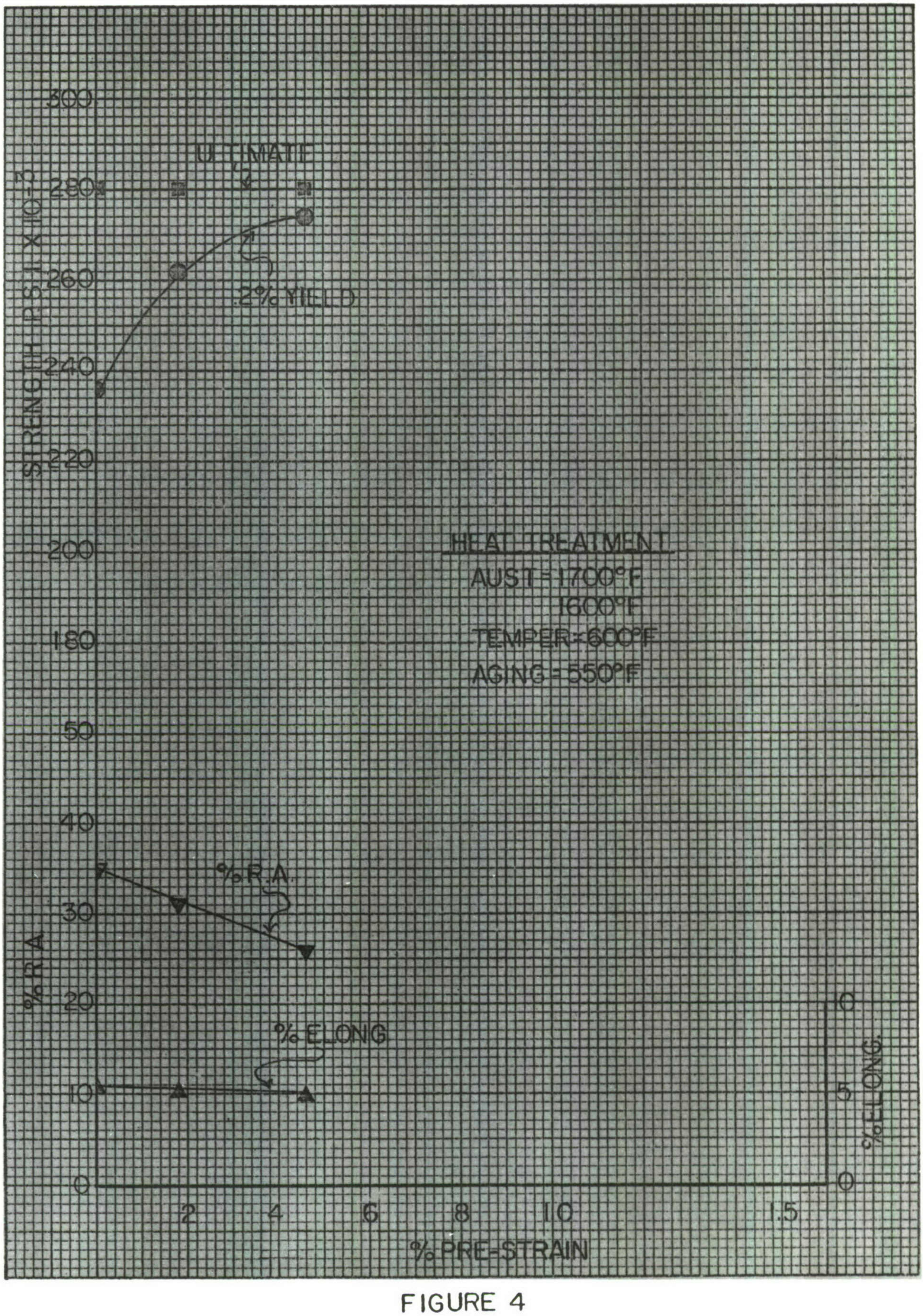



FIGURE 4
TENSILE PROPERTIES OF MAR-STRAINED
300M

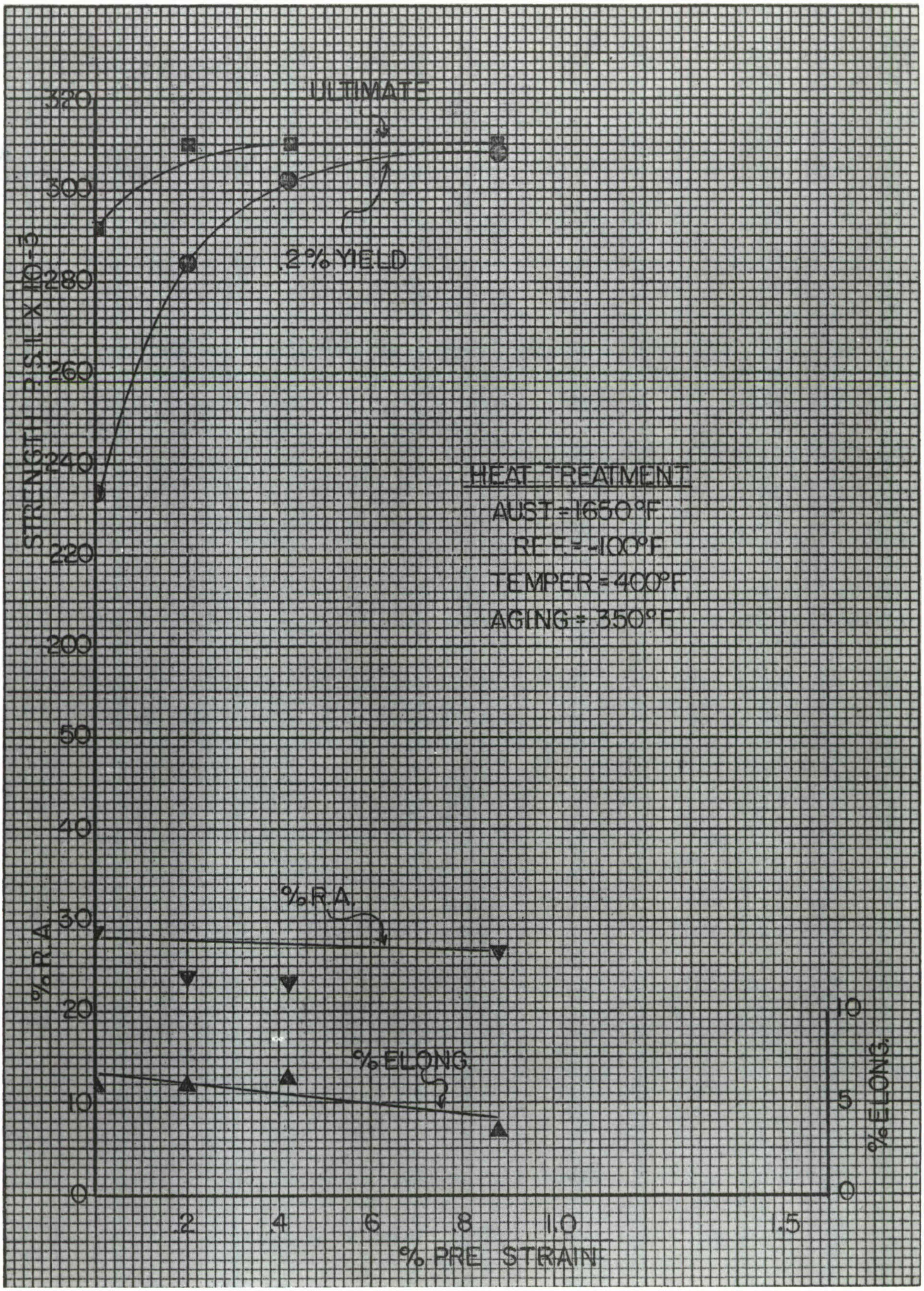



FIGURE 5

TENSILE PROPERTIES OF MAR-STRAINED WCM-4B

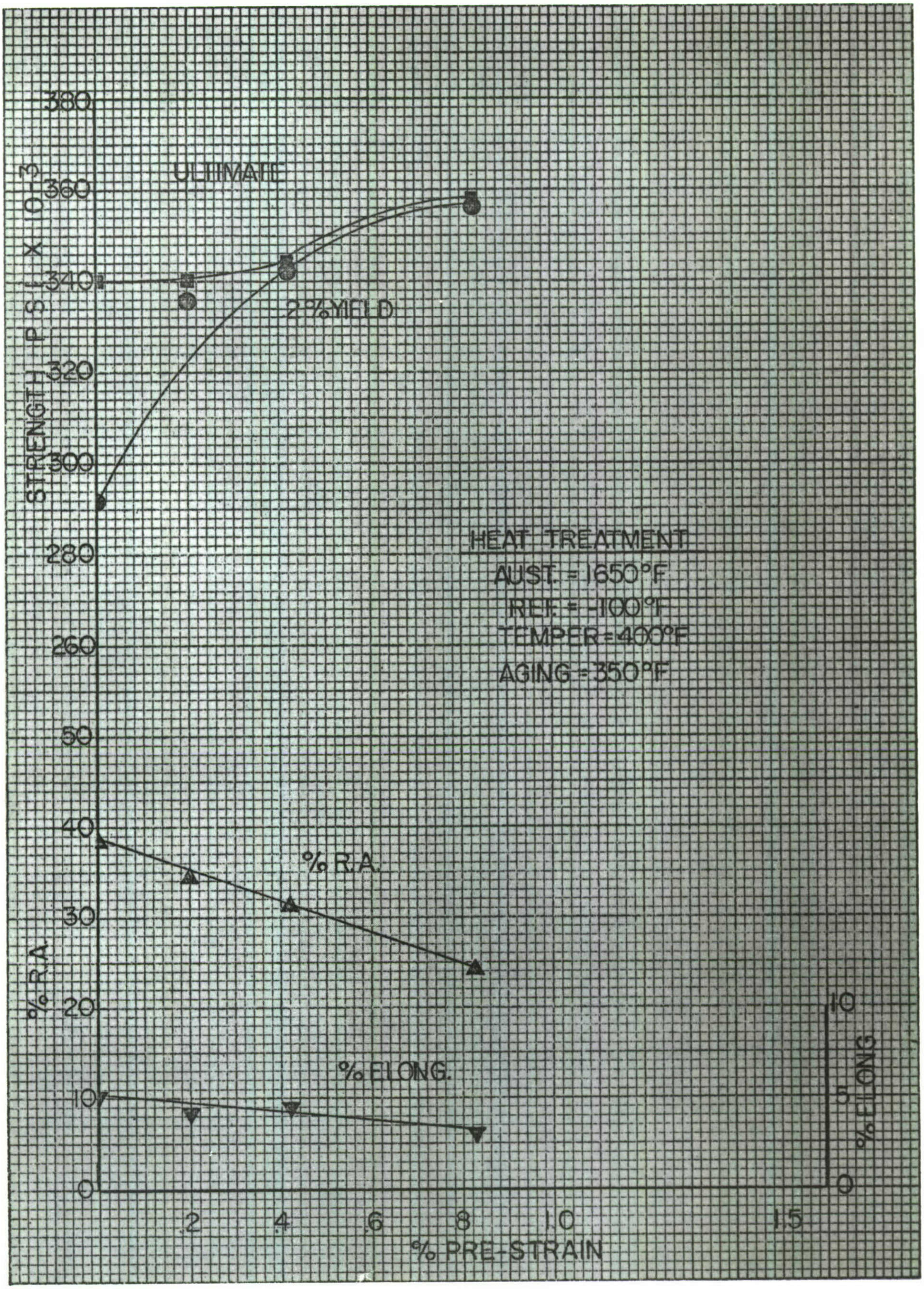



FIGURE 6

TENSILE PROPERTIES OF MAR-STRAINED

WCM-4B

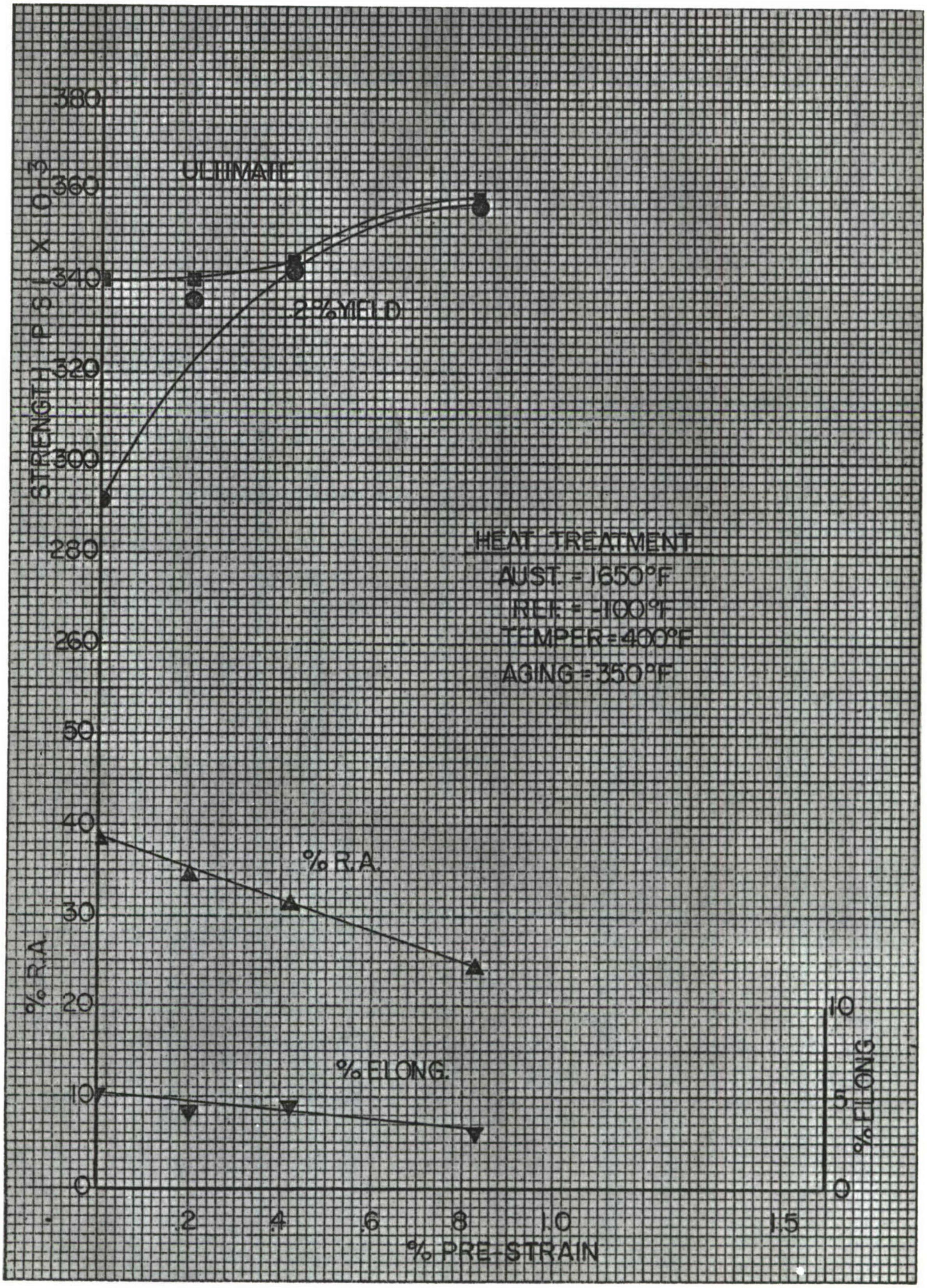



FIGURE 7

TENSILE PROPERTIES OF MAR-STRAINED

WCM-1B

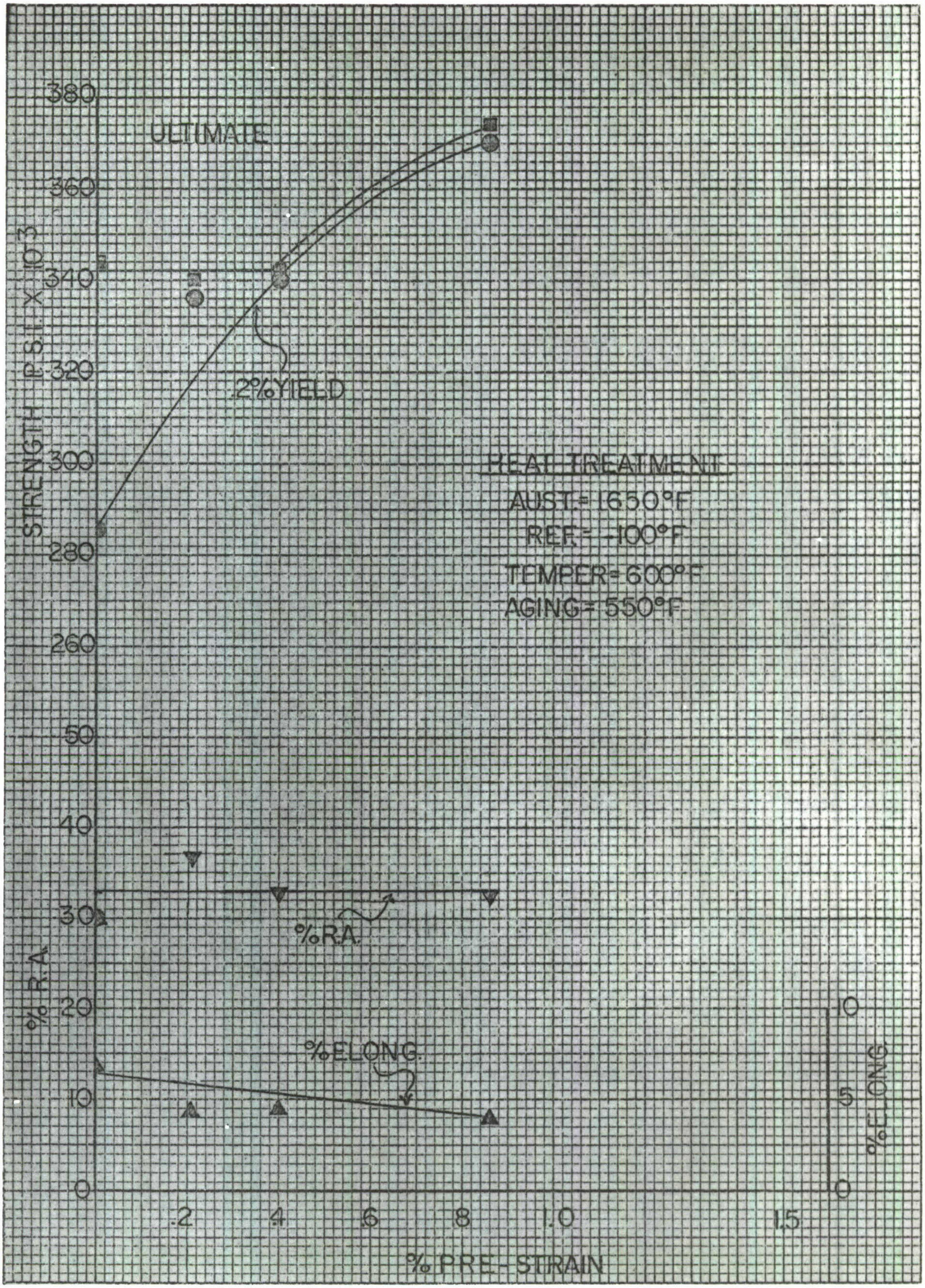



FIGURE 8
TENSILE PROPERTIES OF MAR-STRAINED
WCM-IB

4. Ductility, as measured by reduction in area and elongation, was not appreciably affected by Mar-Straining when pre-straining was less than about 0.5%.

Also investigated was the effect of aging time and temperature on the amount of strength increase. Several 300M specimens (tempered at 600°F) were pre-strained 0.4% (optimum amount of strain in preliminary testing) and were aged at room temperature, 212, 300, 400, 500 and 700°F. A constant aging time of four hours was maintained. These specimens were then tested and the percentage increase between the final stress encountered during the pre-straining and the new Mar-Strain 0.02% yield calculated. These data, plotted in Figure 9, show that a temperature of at least 300°F was required to produce a significant aging effect. After 300°F aging, an increase of 10% in the Mar-Strained yield point over the stress required to achieve 0.4% pre-strain was obtained. This 10% increase was nearly constant between 300°F and the original 600°F tempering temperature. The 700°F age showed a slight drop in percent increase to about 7%. Aging at room temperature produced no increase in the new yield point over the pre-straining stress and was equivalent to an interrupted tensile test.

In order to determine the effect of time of aging, several bars were strained 0.4% and aged at 400°F for varying lengths of time. These results are shown in Figure 10. Very little decrease in percentage was encountered with time. An aging time of one hour was all that was required to achieve maximum increase in properties.

After establishing optimum aging time and temperature, the effect of varying amount of pre-strain on 300M was again determined. An aging time of two hours at a temperature of 400°F was used. The results are shown in Figure 11. These data, contrasted with the original data obtained, demonstrated the advantages of optimum aging.

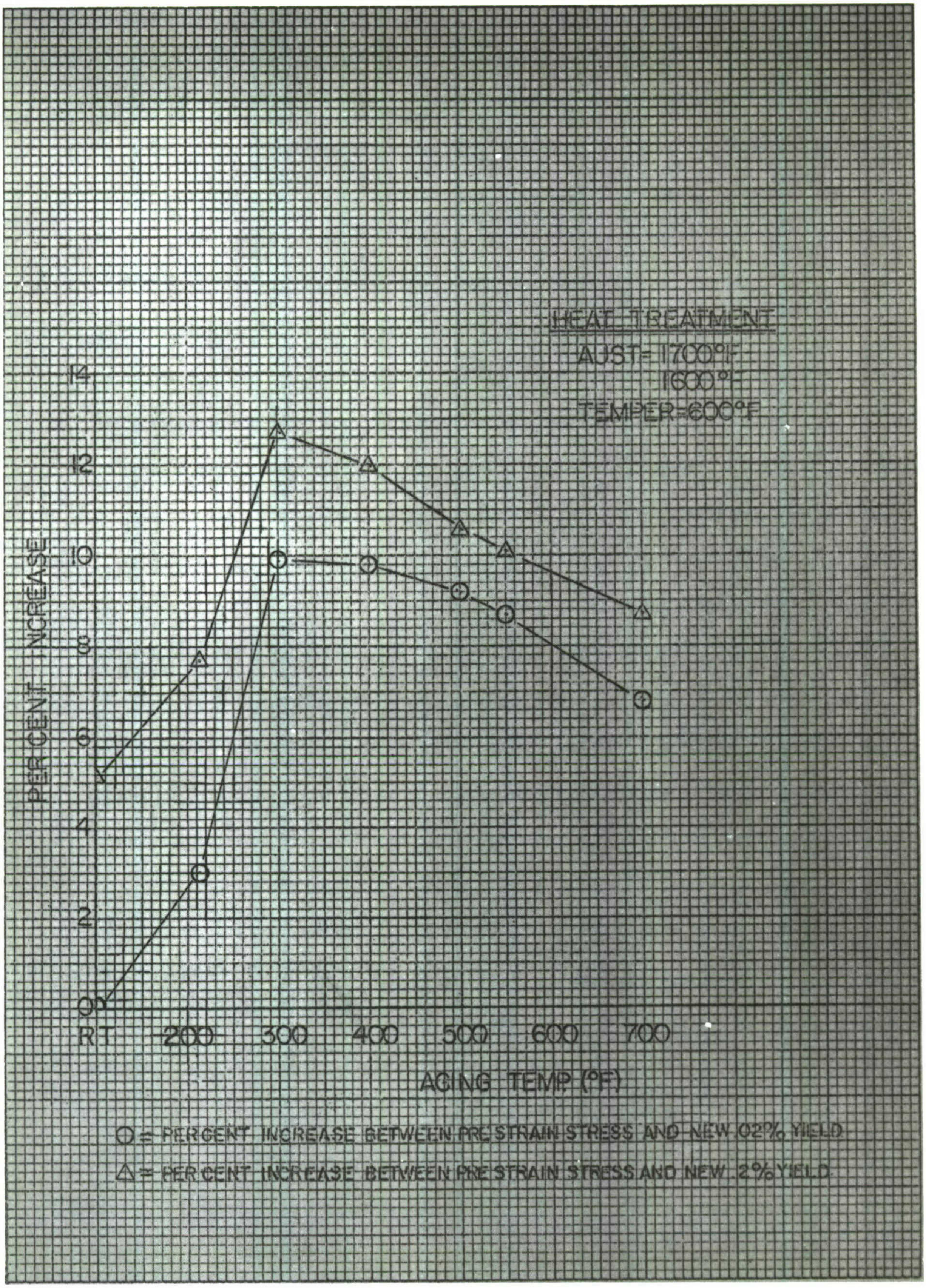



FIGURE 9
EFFECT OF AGING TEMPERATURE ON MAR-STRAINED
300-M

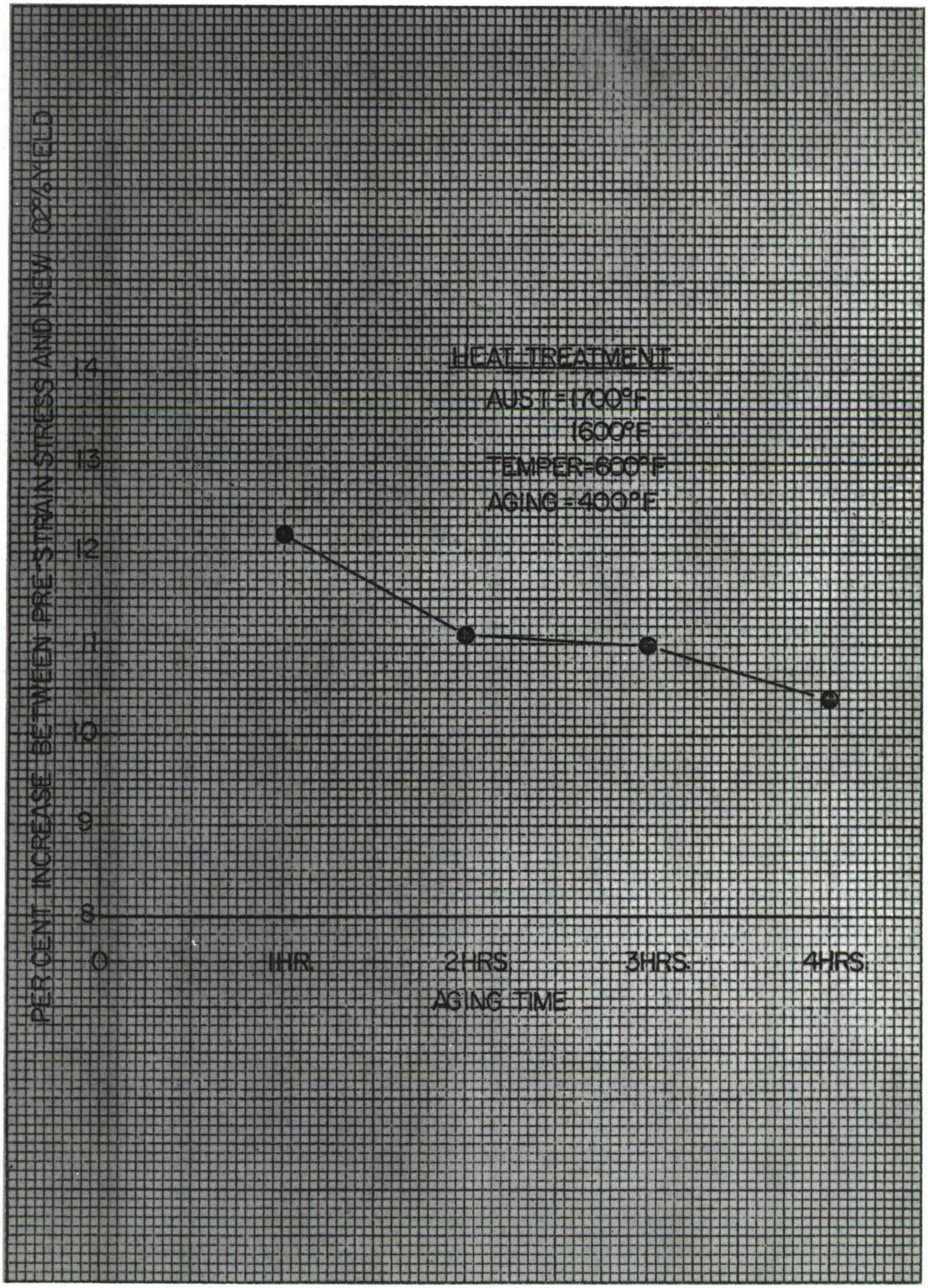



FIGURE 10
EFFECT OF AGING TIME ON MAR-STRAINED
300-M

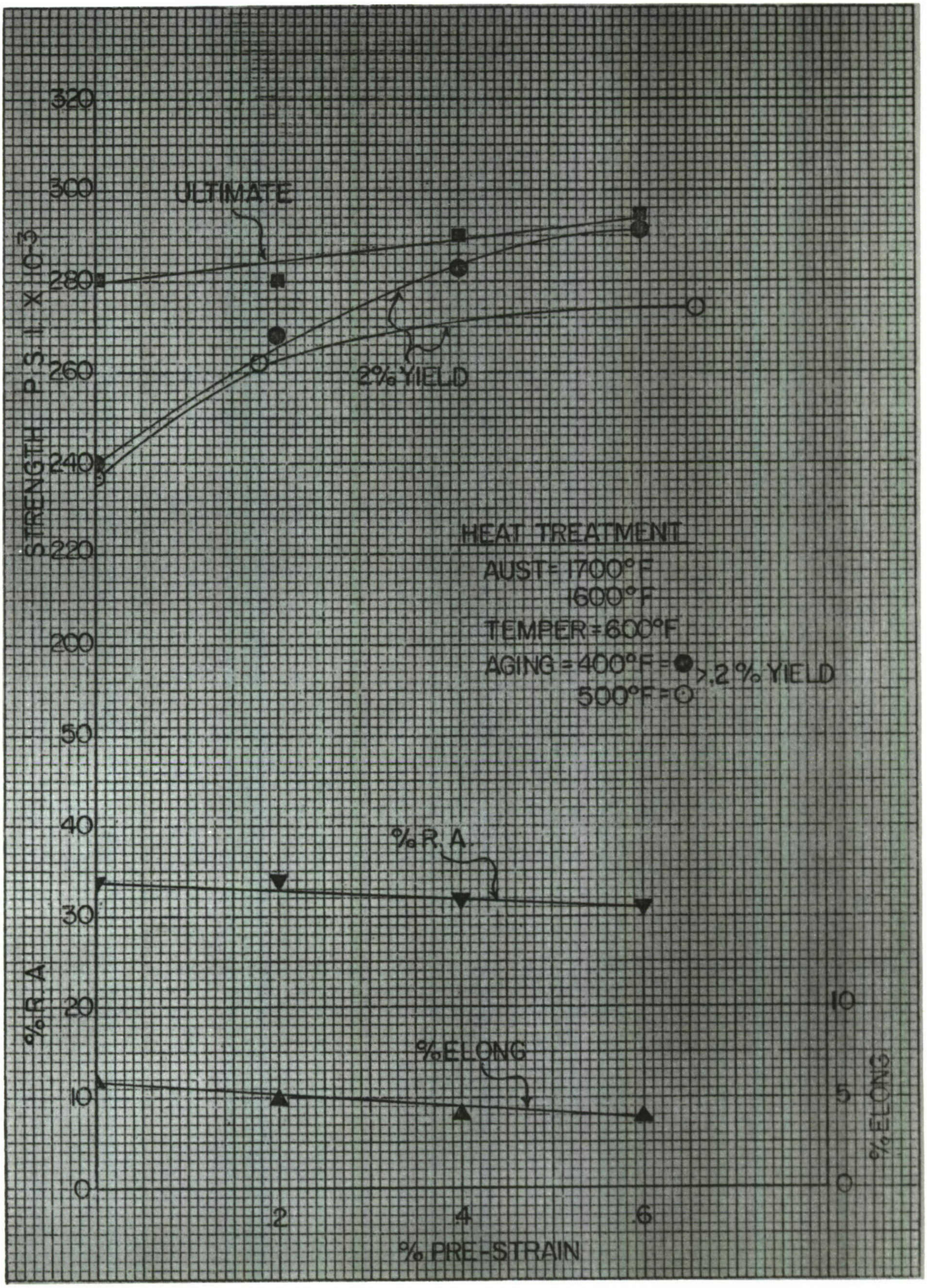



FIGURE II

TENSILE PROPERTIES OF MAR-STRAINED 300-M OPTIMUM AGING

## B.) Procedure

In conjunction with the ASD project engineer, eight (8) alloys were selected to determine their response to the Mar-Strain process. This selection was based on the present utilization in aircraft and missile systems, on their potential use because of their high strength properties, and on their expected Mar-Strain response. The alloys selected, grouped according to their class, were:

1. Low alloy martensitic

   300M
   D6AC
   WCM-4
   WHC
   Mod. S-5

2. Secondary Hardening (H-11)

   Vascojet 1000

3. Semi-Austenitic Stainless

   AM-355

4. Martensitic Stainless

   422

(Chemical compositions are shown in Table 1)

Heat treatments for each alloy were selected based on data available in the literature, unpublished laboratory data, and data supplied by steel vendors. The heat treat selections were based on the highest heat treated strength obtainable which retained sufficient ductility for adequate pressure vessel performance and good strain hardening characteristics. The heat treatments selected for each alloy are shown in Table 2. All specimens were coated with a "No-carb" coating and austenitized in an argon atmosphere to prevent decarburization. Tempering was in air. A standard ASTM sheet type specimen, .500 inch gage width and two inch gage length, was used Thicknesses ranged from .080 to .100 inches.

## TABLE 2

## HEAT TREATMENTS EVALUATED

| Alloy | Austenitizing (1) Temp. °F | Time Hrs. | Quench | Tempering Temp. °F | Time Hrs. | Aging Temp. °F | Time Hrs. |
|---|---|---|---|---|---|---|---|
| 300M | 1650 | 1/2 | Salt (2) | 400 | 2 + 2 | 350 | 2 |
| | | | | 600 | 2 + 2 | 400 | 2 |
| Ladish D6AC | 1650 (NOR) | 1/2 | | | | | |
| | 1550 (Aust) | 1/2 | Salt | 400 | 2 + 2 | 350 | 2 |
| | | | | 600 | 2 + 2 | 300 | 2 |
| | | | | 800 | 2 + 2 | 400 | 2 |
| WCM-4 | 1650 | 1/2 | Air | 600 | 2 + 2 | 400 | 2 |
| Mod S-5 | 1650 | 1/2 | Salt | 575 | 2 + 2 | 400 | 2 |
| WHC | 1650 | 1/2 | Air | 600 | 2 + 2 | 400 | 2 |
| Vascojet 1000 | 1850 | 1/2 | Air | 1000 | 2 + 2 | 600 | 2 |
| | | | | 1000 | 2 + 2 | 800 | 2 |
| AM-355 | 1850 | 1/2 hr. | | | | | |
| | 1710 (Cond) | 1/2 hr. | Air | 850 | 2 | 400 | 2 |
| | | | | 850 | 2 | 700 | 2 |
| | | | | 1000 | 2 | 900 | 2 |
| 422 | 1900 | 1/2 hr. | Air | 800 | 2 + 2 | 400 | 2 |
| | | | | 800 | 2 + 2 | 750 | 2 |
| | | | | 980 | 2 + 2 | 400 | 2 |
| | | | | 980 | 2 + 2 | 750 | 2 |

(1) Tensile bars protected from decarburization with "No-Carb" coating and an argon atmosphere.

(2) 350°F

After selection of heat treatments to be studied, the post-strain heat treatment was optimized in order to utilize to the fullest extent the response of each alloy to the Mar-Strain process. Finally, the effect of varying amounts of pre-strain was determined for each alloy. In summary, the procedure followed in screening each alloy for Mar-Strain response can be outlined as follows:

1) Selection of heat treatment
   a. Austenitizing and quenching
   b. Tempering conditions
2) Optimization and selection of post-strain heat treatment
   a. Constant amount of pre-strain (0.4%)
   b. Aging temperature
   c. Aging time
3) Determination of the effect of pre-strain on the tensile properties using an optimized aging cycle.

During this tensile evaluation, the amount of pre-strain was controlled with a variable transformer extensometer. The amount of pre-strain was taken as the offset measured from the straight line portion (elastic) of a load-extension curve. Elongation measurements reported were those obtained on Mar-Strained tensile specimens. Gage marks were not made until after the pre-straining and aging. Width and thickness measurements were taken after Mar-Straining to calculate the new stresses and the reduction in area; therefore, the percents reported were representative of Mar-Strain ductility.

The aging response of each alloy class was assessed using a constant amount of pre-strain, 0.4%, and varying aging temperatures, at least four

temperatures, between room temperature and the original tempering temperature. Constant times of one hour and two hours were also used except for room temperature aging which varied from 16 to 24 hours. Complete aging response was determined for each alloy class using only one alloy chosen as representative. The response was determined on the following alloys.

1. Low alloy martensitic - D6AC plus pre-contractual data on 300M
2. Secondary hardening martensitic - Vascojet 1000
3. Semi-austenitic stainless - AM355
4. Martensitic stainless - 422

To demonstrate the direct benefit of the aging reaction, the percentage increase between the final stress encountered in pre-straining to 0.4% and the new 0.02% and 0.2% yield strength obtained on re-testing was calculated. This procedure is shown schematically in Figure 12. The calculation of percentages allowed some variations in strength because of scatter in properties caused by the original heat treatment and also allowed comparison of response between alloys within the same class or different classes which were capable of heat treatment to various strength levels.

## C.) Results

### Aging (0.4% Pre-strain)

The results of aging the four alloys chosen as representative of the four classes of steel are shown in Figures 13 through 16. The aging response was obtained for at least two temper conditions of each alloy and represented tempering temperatures between 400°F and 1100°F; consequently, metallurgical structures representative of various stages of martensitic decomposition were encompassed in the study. The martensitic decomposition

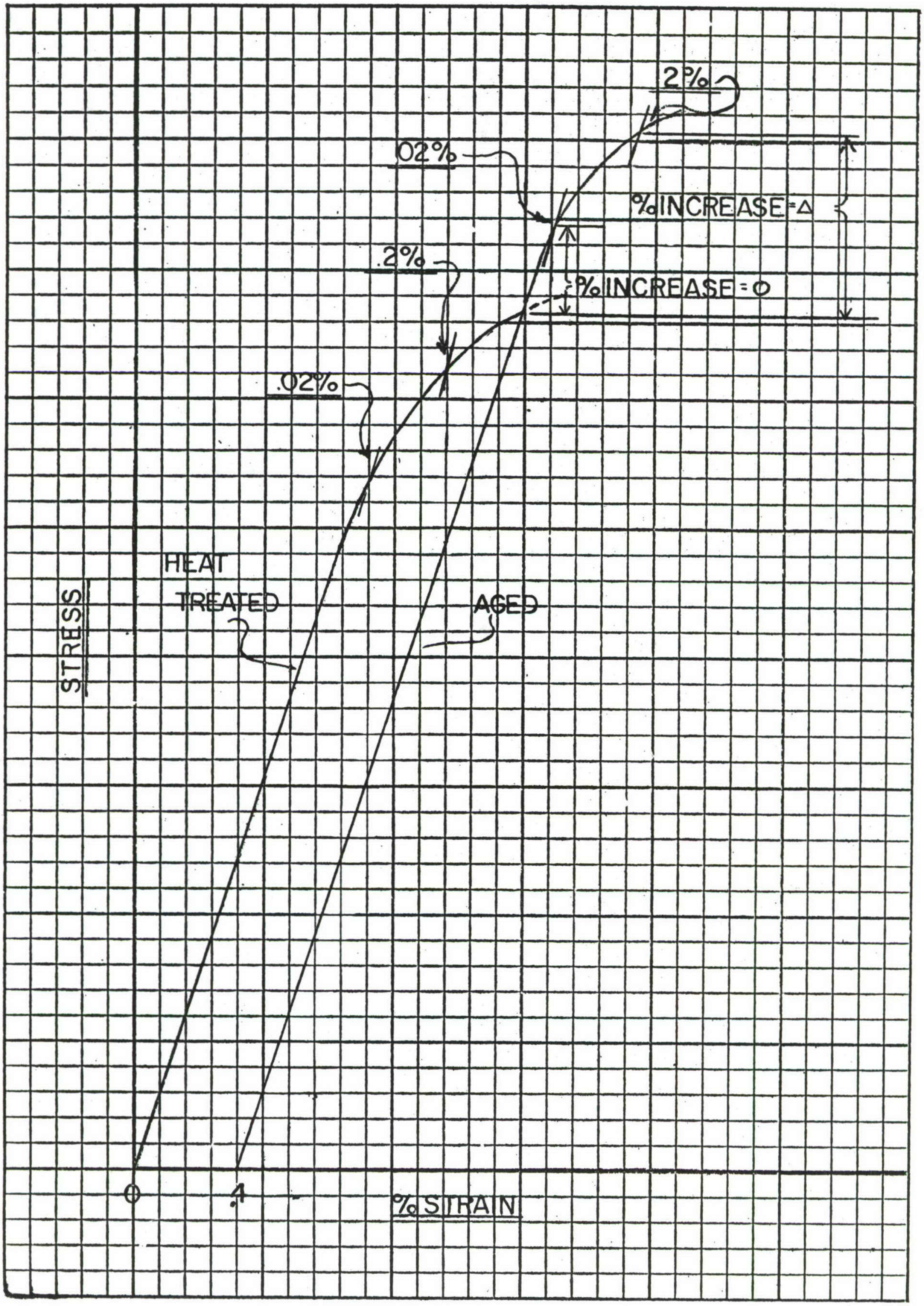



FIGURE 12 EFFECT OF AGING ON TENSILE STRESS-STRAIN CURVE

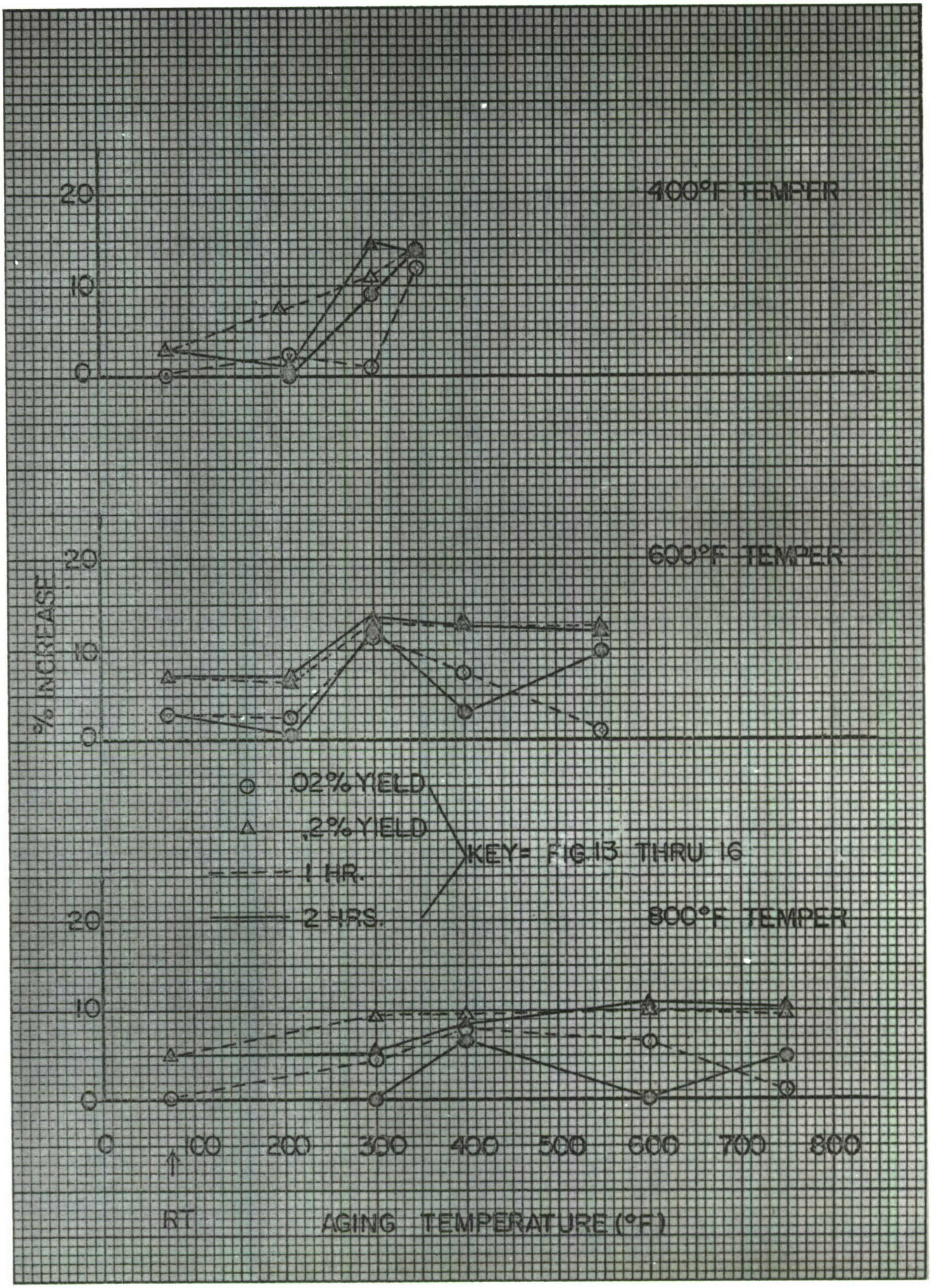



FIGURE 13
EFFECT OF AGING TEMPERATURE ON THE PROPERTIES
OF LADISH D6AC

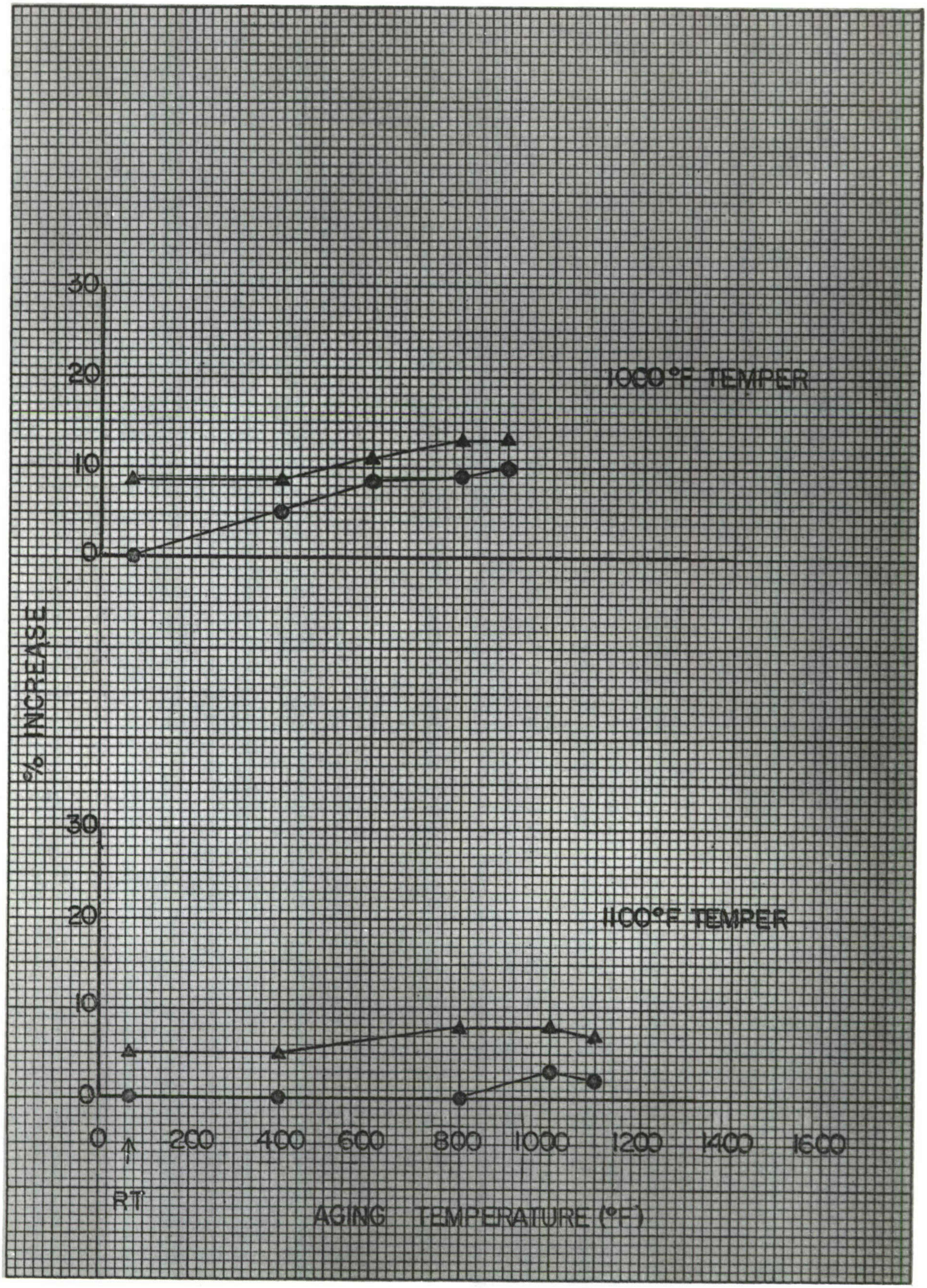



FIGURE 14
EFFECT OF AGING TEMPERATURE
ON VASCOJET 1000

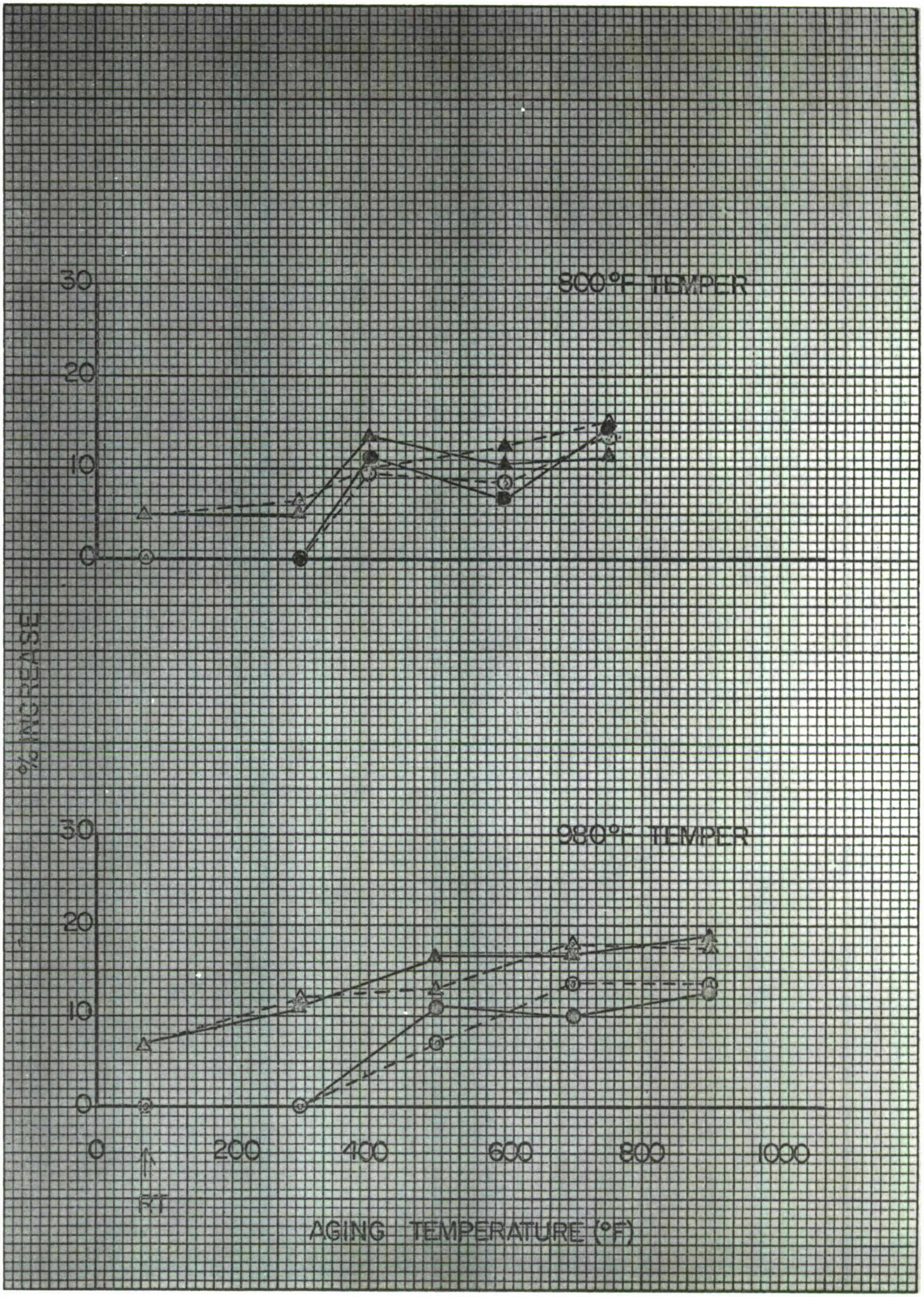



FIGURE 15

EFFECT OF AGING TEMPERATURE

ON 422 SS

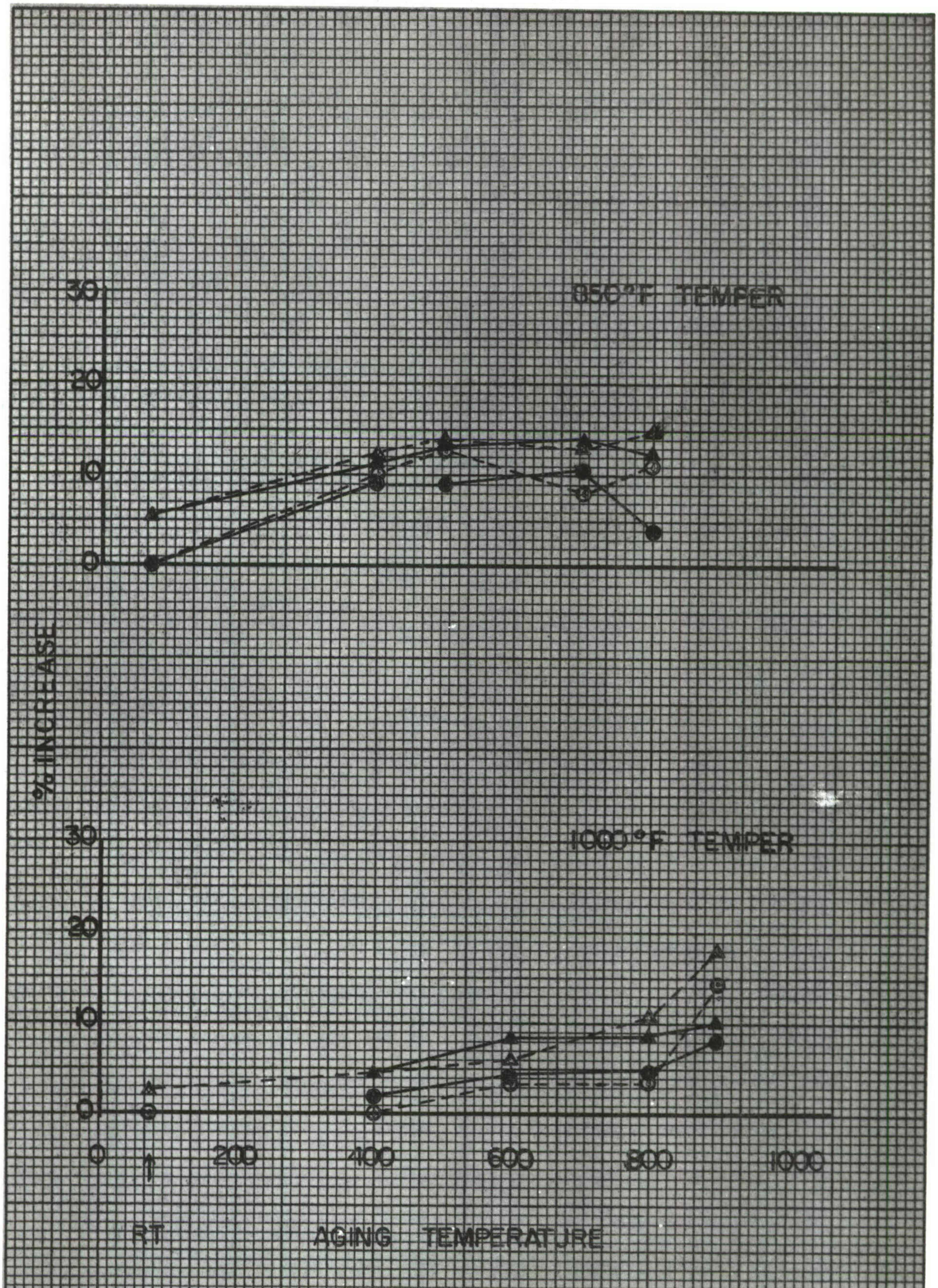



FIGURE 16
EFFECT OF AGING TIME AND
TEMPERATURE ON AM-355

which has been previously discussed was true for all alloys studied with the exception of AM-355 which substitutes nitrogen for carbon to obtain its properties. The precipitation of iron nitrides from martensite follows the same basic metallurgical sequence in that a metastable nitride is precipitated prior to the formation of the more stable $Fe_4N$. The precipitation of this nitride occurs at very nearly the same temperature range as the $Fe_3C$, cementite.

It was noted from examination of these aging curves that the higher the original tempering temperature, the higher the aging temperature required to show an effect from aging. This effect was easily seen by examination of the percent increase in the .02% yield over the pre-strain stress. Still higher aging temperatures were required to produce the maximum increase possible with aging, using the maximum time employed in this study, two hours. This demonstrated the dependency of the aging reaction on the metallurgical structure produced during the initial tempering. For example, as the tempered structure became more stable by precipitation of cementite or complex carbides and nitrides, higher aging temperatures were required to cause migration to and/or precipitation at the pre-strain induced dislocation sites to cause blocking of their movement on subsequent testing. Also, when only metastable compounds such as epsilon carbide are present, as in D6AC tempered at 400 and 600$^{\circ}$F, low aging temperatures of the order of 300 to 350$^{\circ}$F showed maximum increases in yield. The highest tempering temperature studied, 1100$^{\circ}$F, (Vascojet 1000) had almost negligible response to aging, indicating that when the prior structure has been sufficiently stabilized by agglomeration and complete precipitation, aging conditions of two hours at temperatures up to within 50$^{\circ}$F of the original tempering

temperature were not sufficient. Also,at higher aging temperatures ($1000^{o}F$ or greater),annealing of the dislocations produced by pre-strain is competing with the aging reaction.

Further interpretation of the results shown in Figures 13 through 16 is required. The percent increase of the .02% yield strength over the pre-strain stress was the value which was most indicative of the strength of the aging reaction. This value was also subject to a greater amount of spread due to the inaccuracies which are associated in measuring this small an offset with the extensometer used. The percentage increase in .2% yield strength was not only affected by the aging reaction,but was also increased by the natural strain hardening of the heat treated alloy; therefore,the value shown was the sum total of the two processes except where the percentage increases for .02 and .2% yield strengths were equal (usually associated with optimum aging).Then the aging reaction overpowered the strain hardening portion of the .2% value. The relationship of the increase in .2% yield strength and the strain hardening of the alloy will be further explored later.

In summary, all four of the alloy classes responded to the aging treatments. The magnitude of the response was extremely dependent on the as-tempered structure. The more stable the as-tempered structure,the higher the aging temperature required to promote aging to effect maximum increase in yield strength. Although time at aging temperatures was not investigated fully, it is suspected that in some cases longer times can be substituted for increased temperature. The magnitude of the increase in yield was also dependent on the strain hardening characteristics of the alloy. The aging time and temperatures selected for each alloy to determine its Mar-Strain

strength are listed in Table 2. In some cases ,less than optimum aging conditions were used to verify some of the above results.

Strength

Figures 17 through 34 show the effect of various amounts of pre-strain on the alloy heat treated and aged as tabulated in Table 2. These results presented several conclusions which were common among all alloys studied under optimum conditions.

1) The greater part of the increase in .2% yield was achieved with as little as .4% pre-strain.
2) The ultimate strength of these alloys, as Mar-Strained, remained nearly unaffected up to about .5% plastic pre-strain. At this point, when specimens containing more than this amount were tested, the new yield and ultimate strength were equal and were increased concurrently with the amount of pre-strain.
3) When low amounts of pre-strain were used, Mar-Strain ductility, as measured by percent reduction in area and elongation, was not appreciably affected.

It has been previously discussed that the increase in yield strength was strongly dependent on the strain hardening characteristics of the alloy. This relationship is demonstrated in Figure 35. The ratio of the new .2% yield strength (after pre-straining .4% and optimum aging) over the stress required to obtain .4% pre-strain is plotted versus a measure of the strain hardening of the as heat treated alloy. The measure of strain hardening was obtained by plotting the percent pre-strain versus the stress to obtain this amount of strain on log-log paper and calculating the slope of the resultant straight line. The value of this slope was taken as the measure of

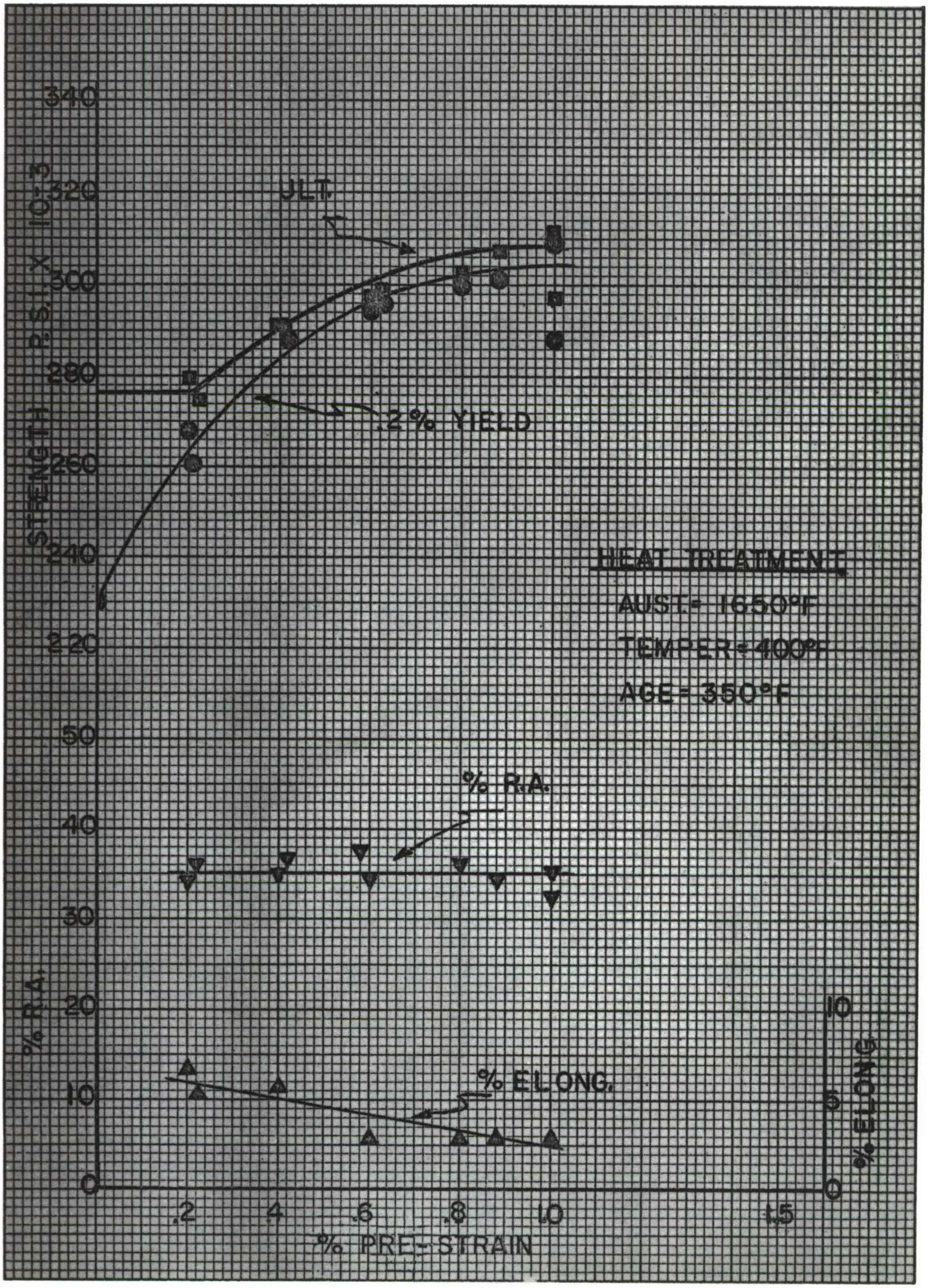



FIGURE 17

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF 300M

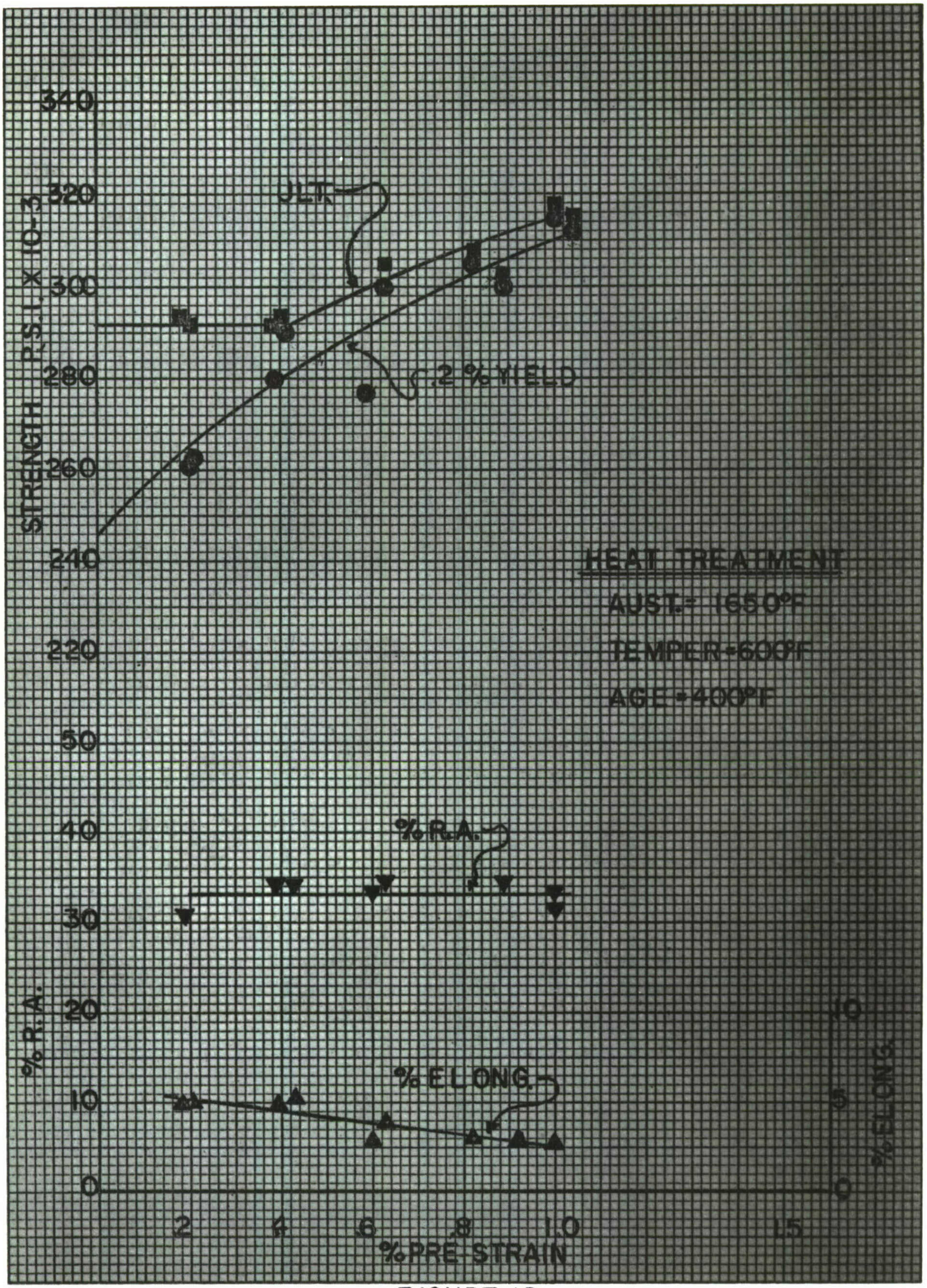



FIGURE 18

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF 300M

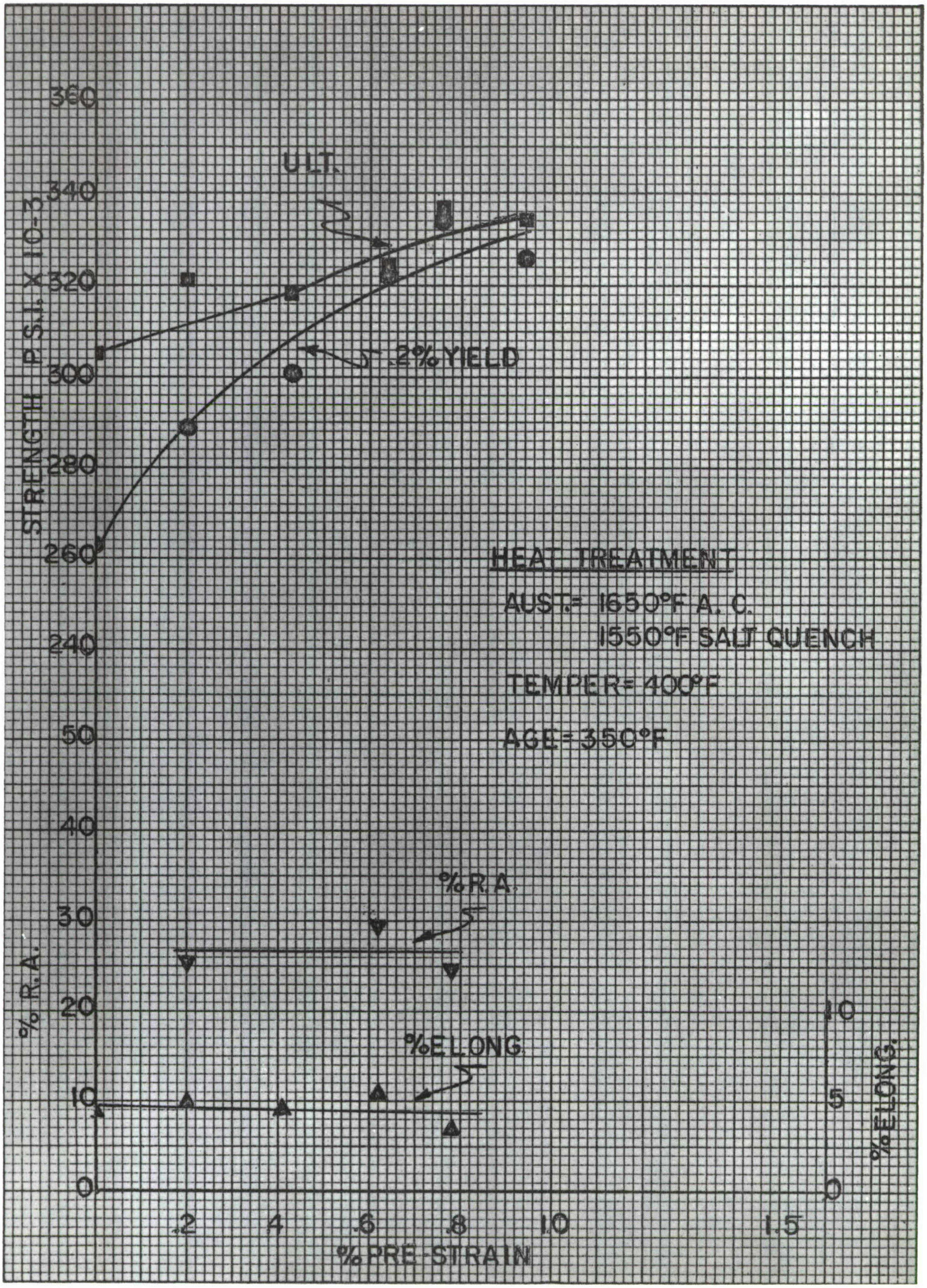



FIGURE 19

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF LADISH D6AC

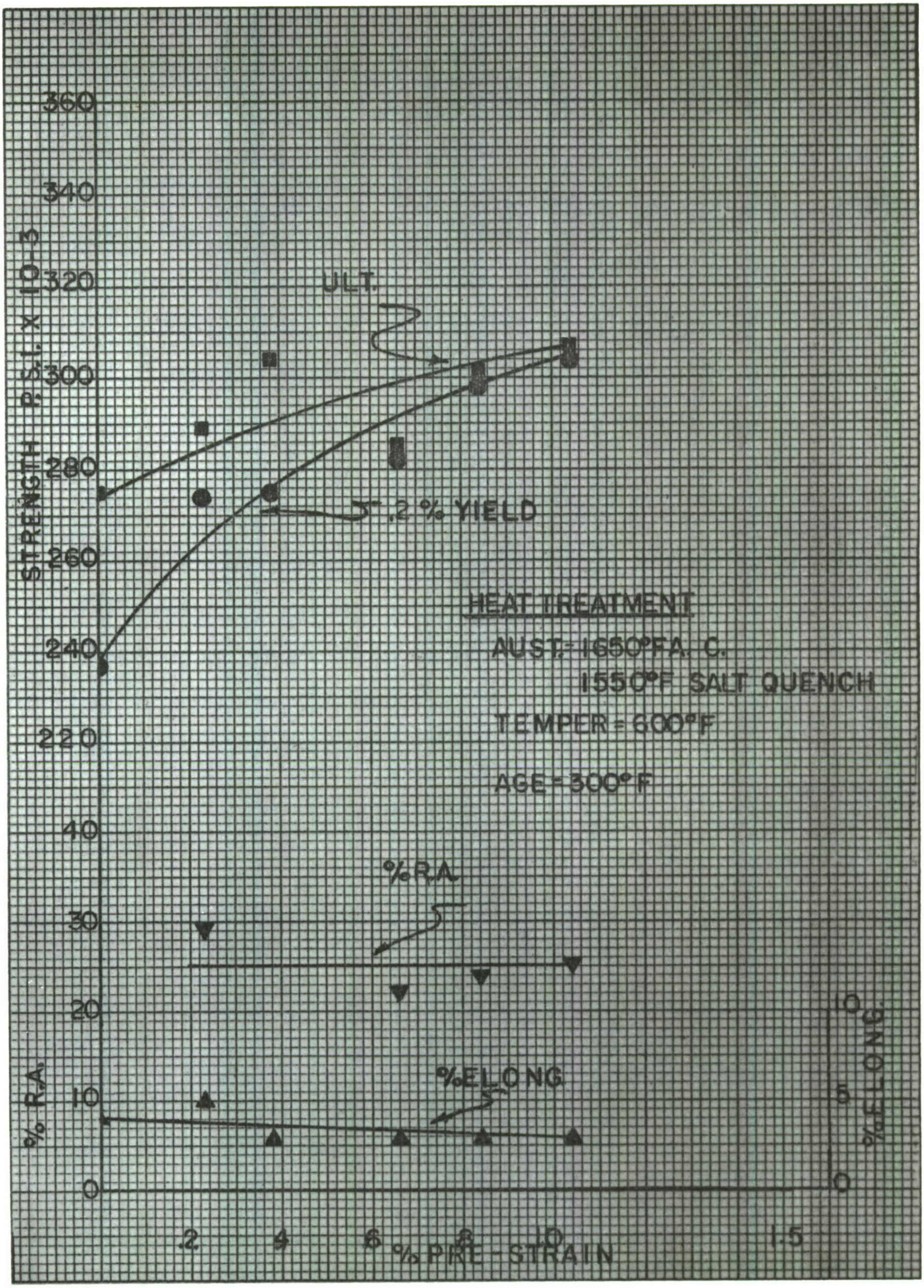



FIGURE 20

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF LADISH D6AC

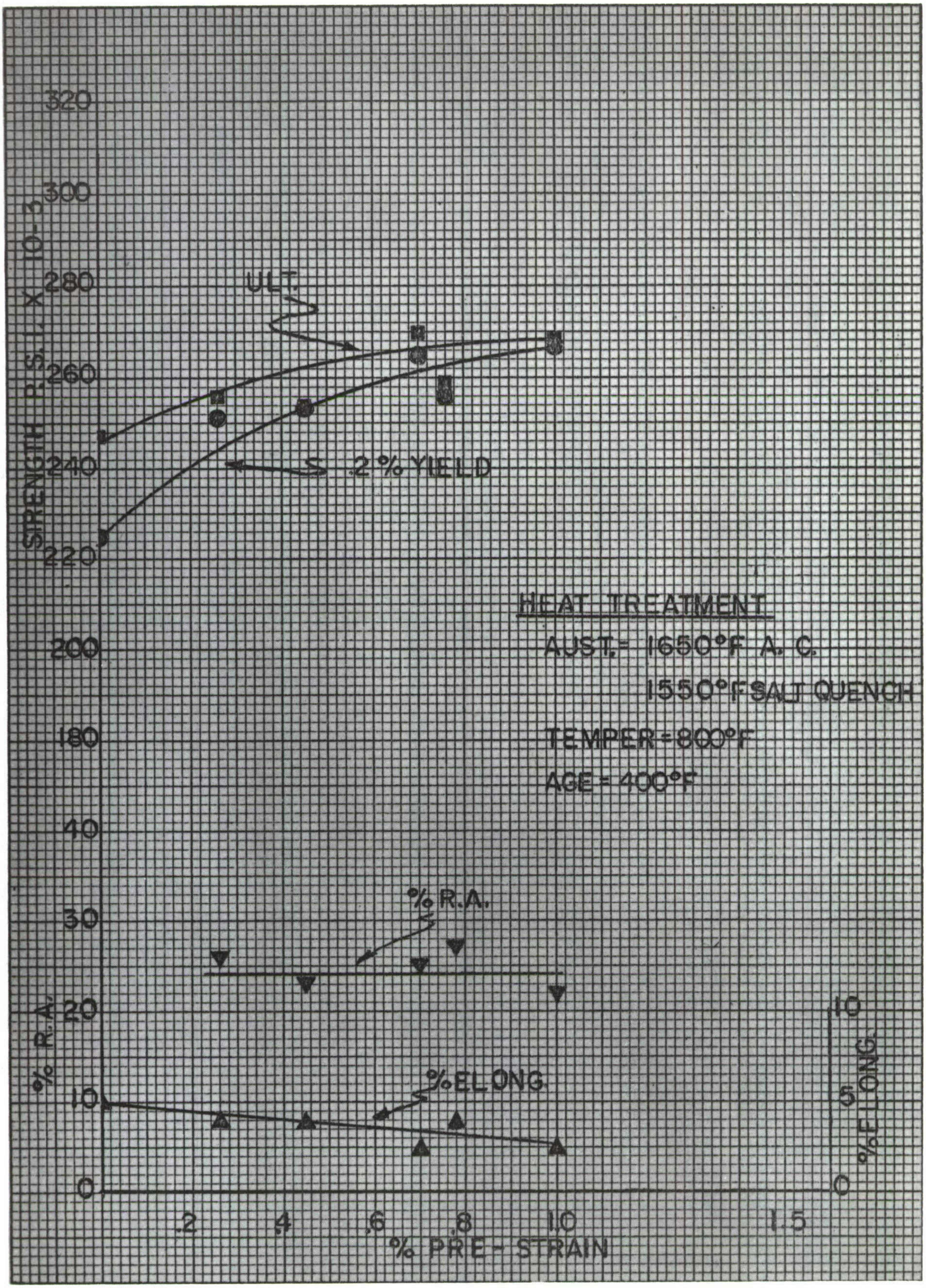



FIGURE 21

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF LADISH D6AC

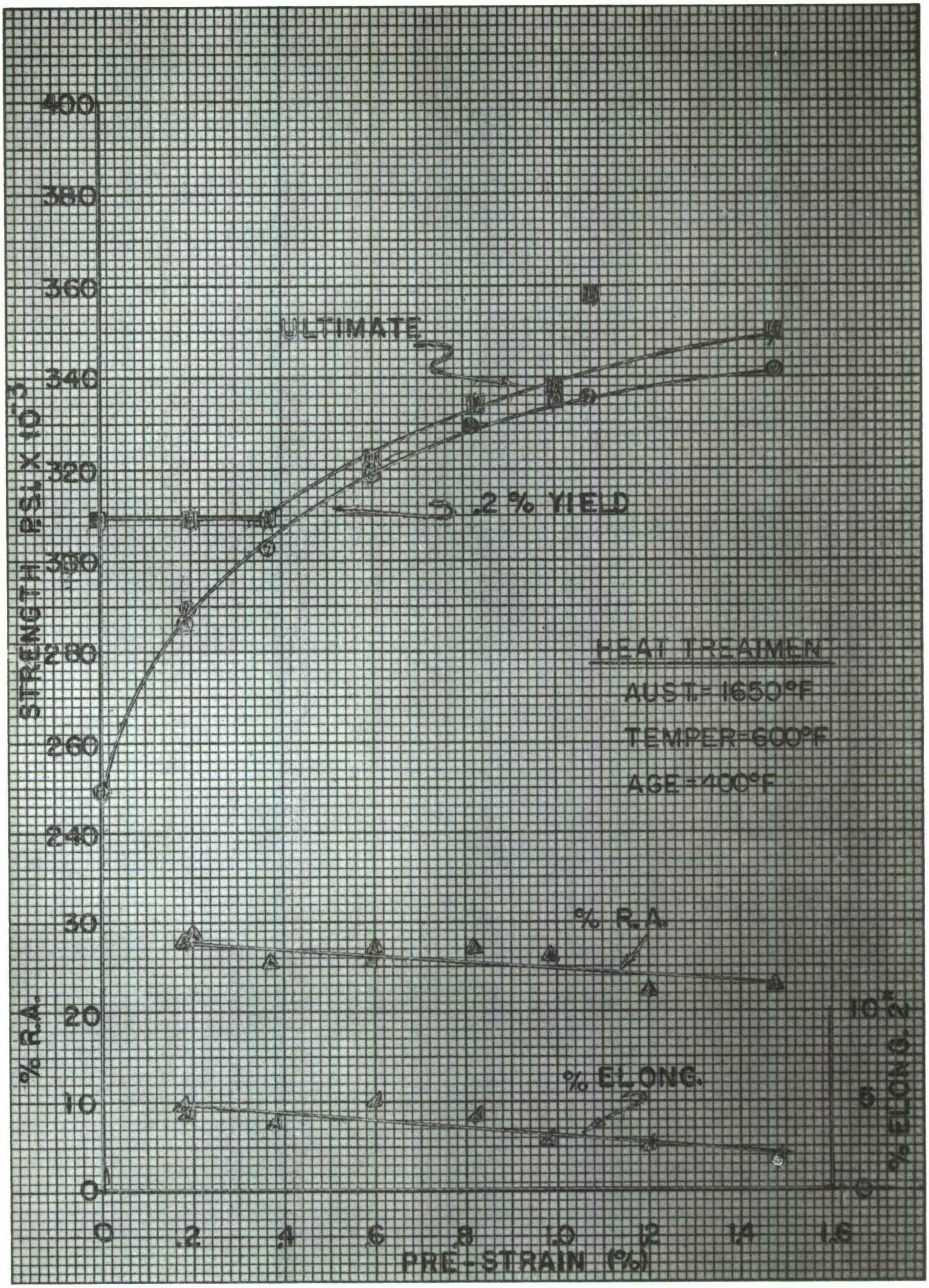



FIGURE 22

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF WCM-4 (REFRIGERATED)

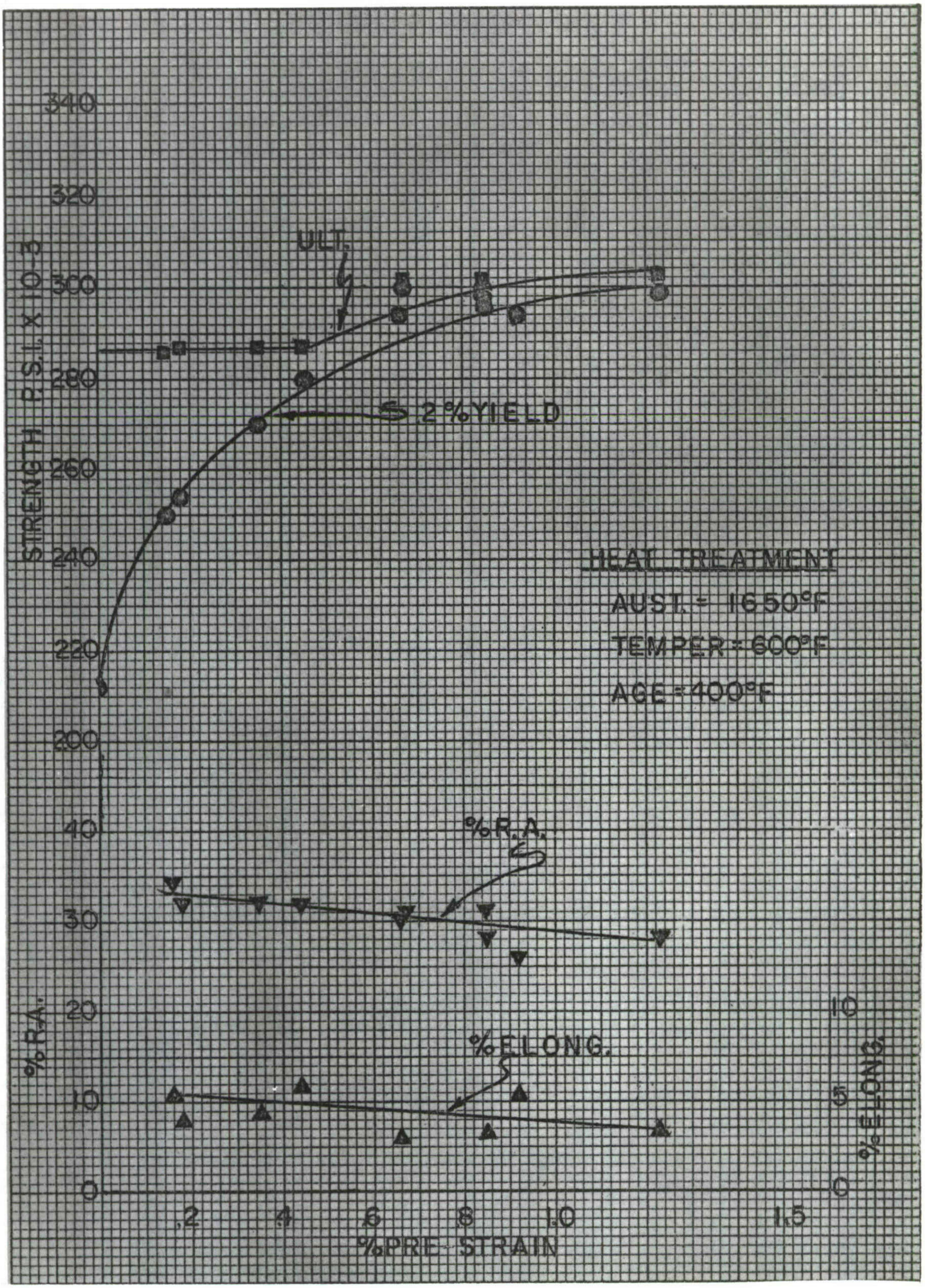



FIGURE 23
EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF WCM-4
(NOT REFRIGERATED)

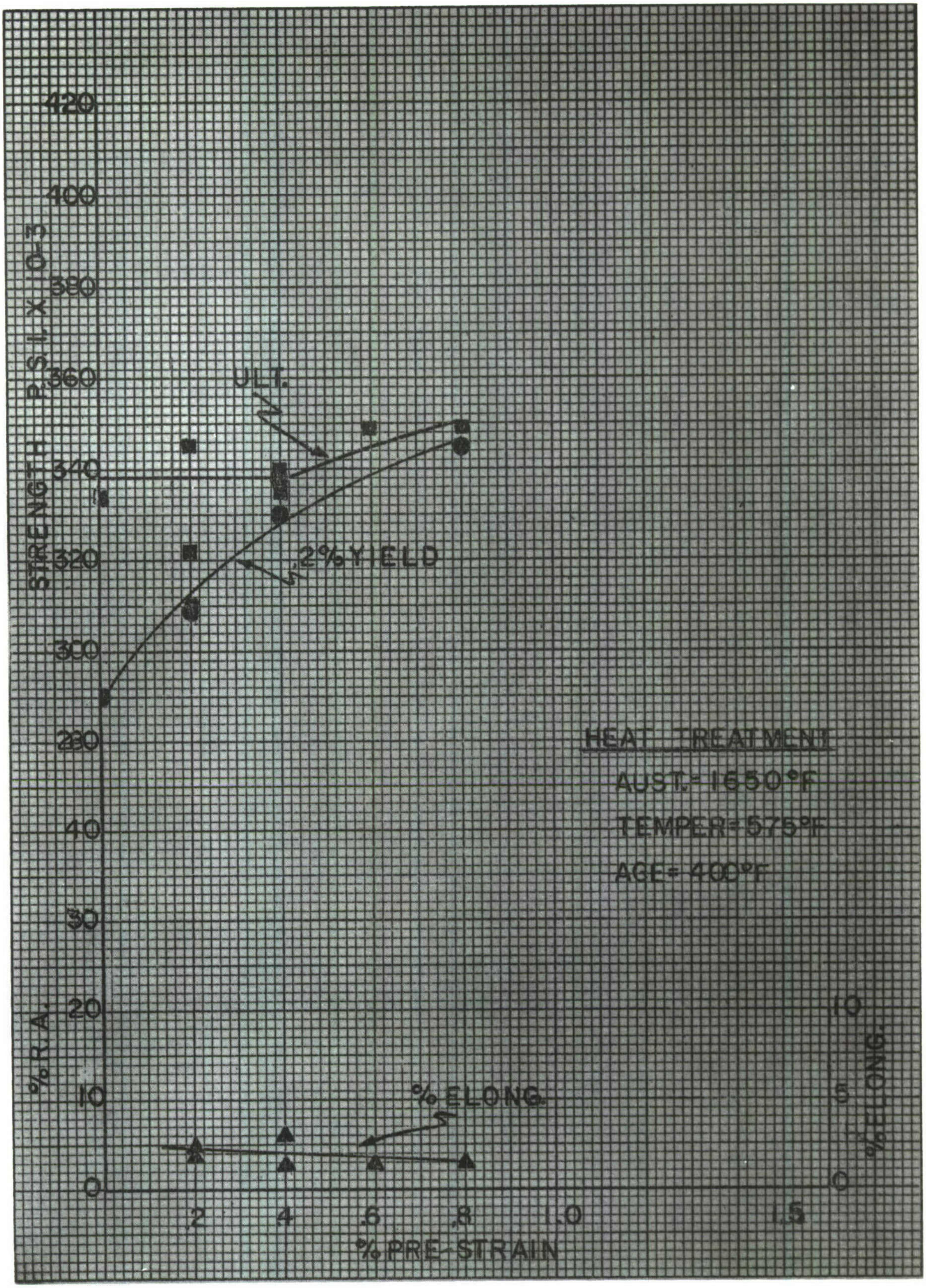



FIGURE 24

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF MOD.S-5

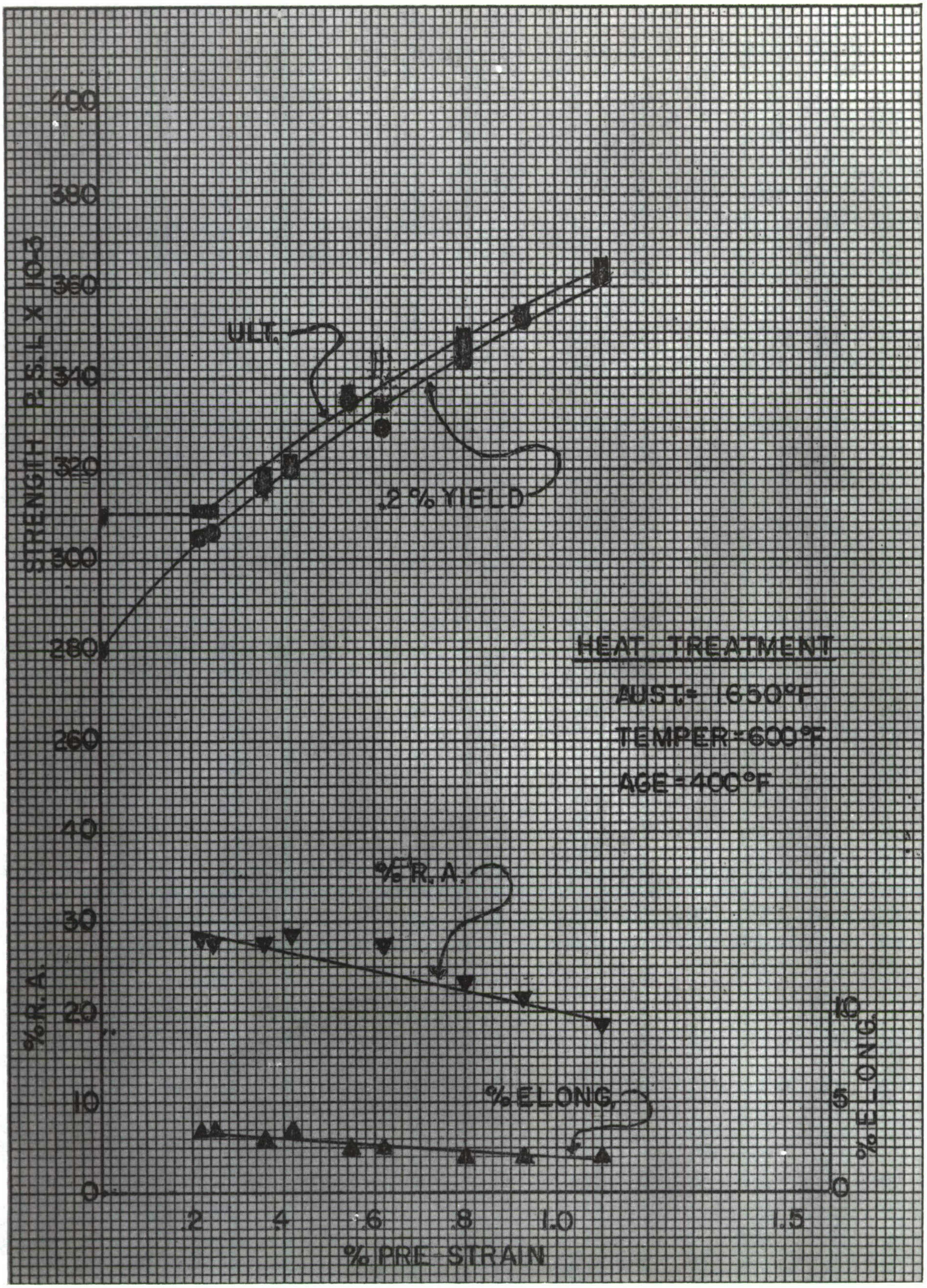



FIGURE 25

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF WHC

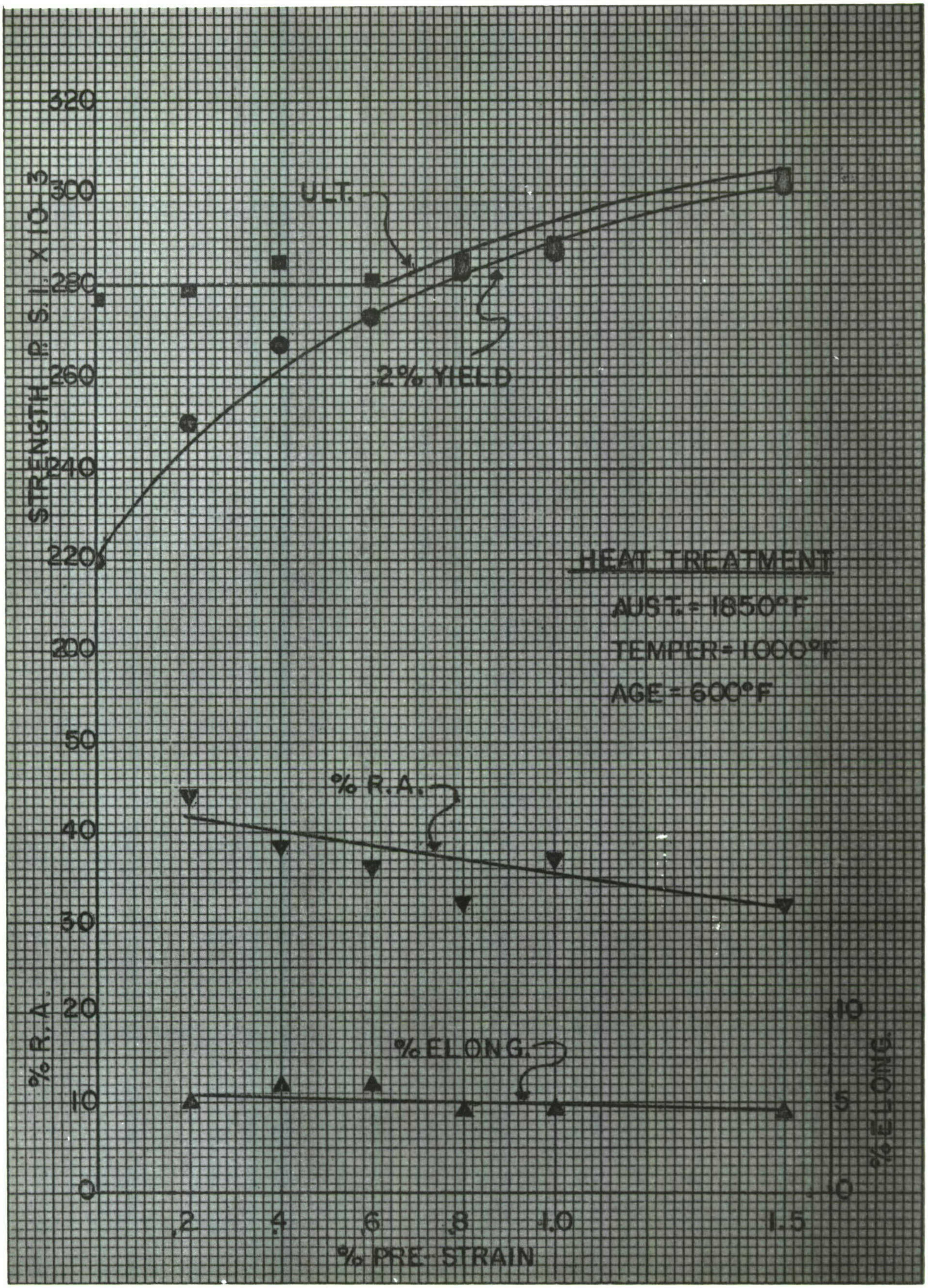



FIGURE 26

EFFECT OF PRE-STRAIN WITH LESS THAN OPTIMUM AGING ON THE PROPERTIES OF VASCO JET 1000

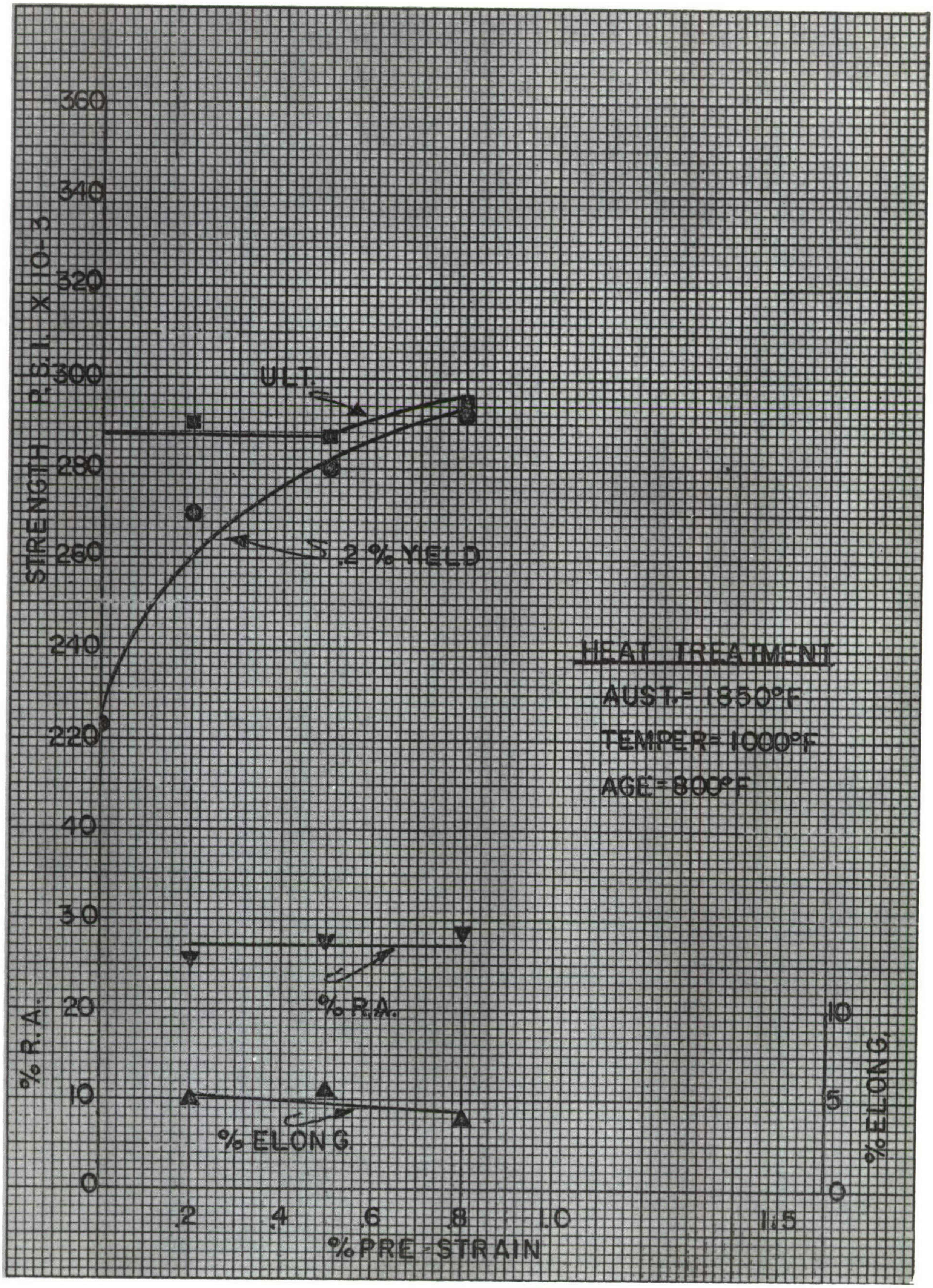



FIGURE 27
EFFECT OF PRE-STRAIN WITH OPTIMUM AGING
ON THE PROPERTIES OF VASCO JET 1000

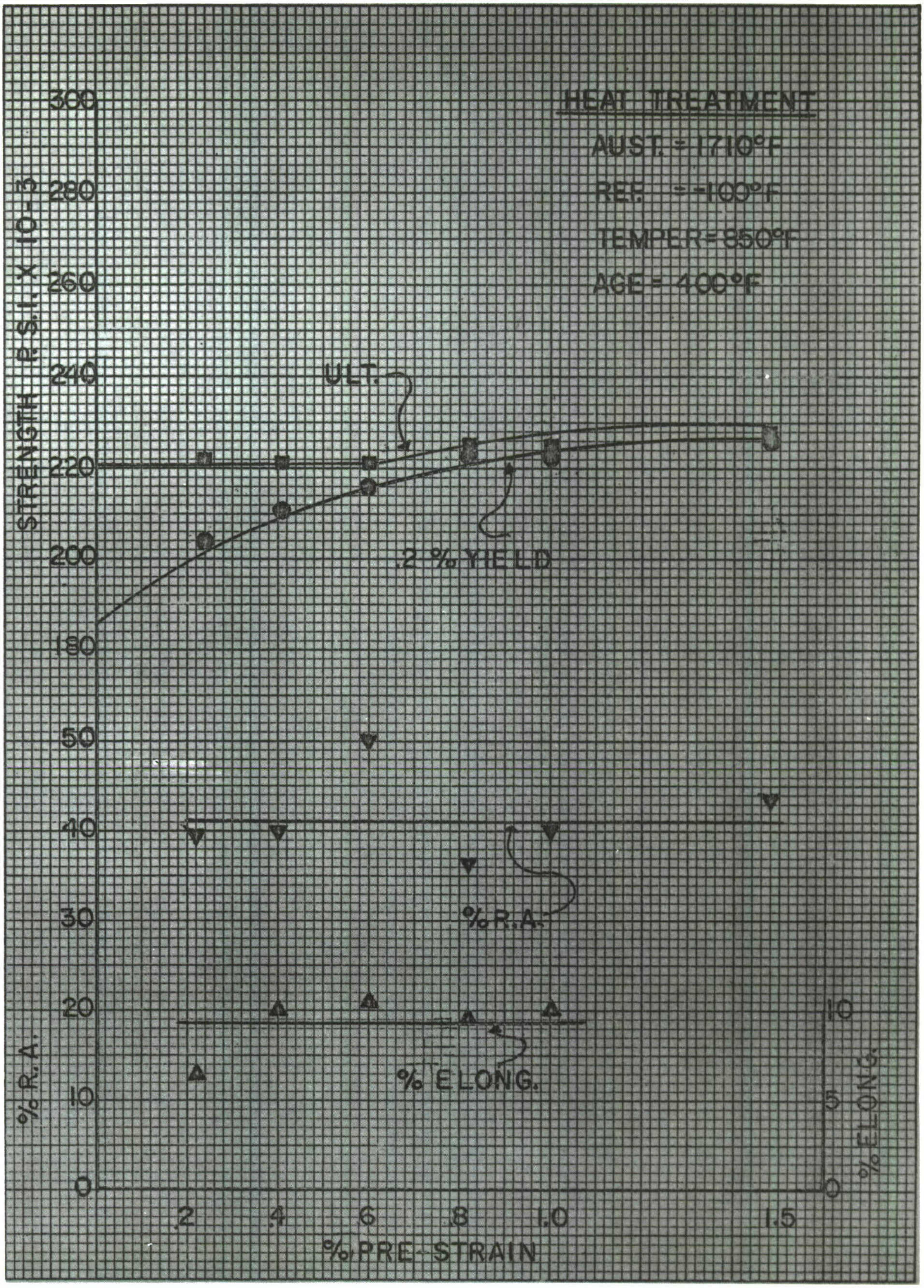



FIGURE 28
EFFECT OF PRE-STRAIN WITH LESS THAN OPTIMUM AGING ON THE PROPERTIES OF AM-355

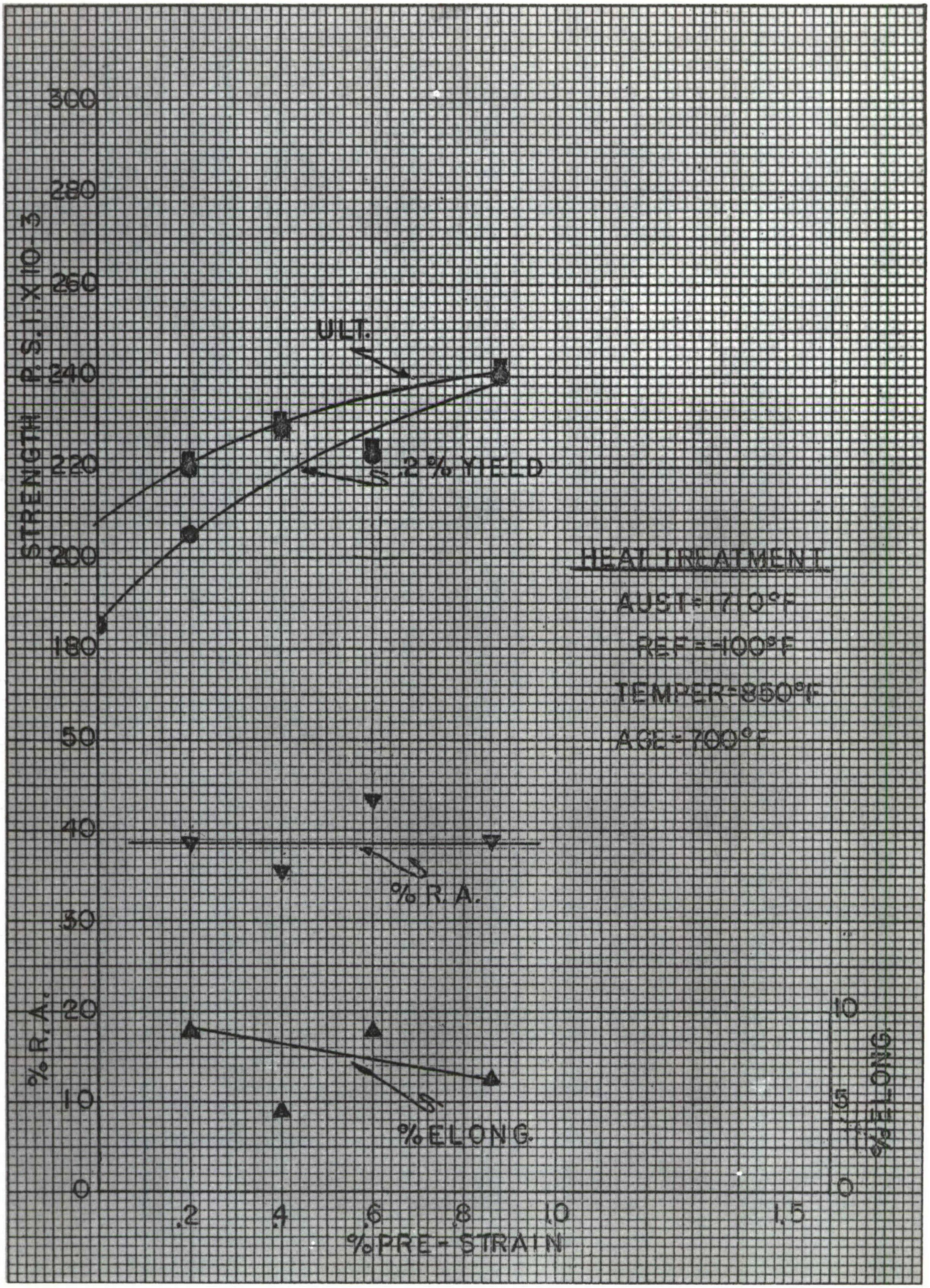



FIGURE 29

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF AM-355

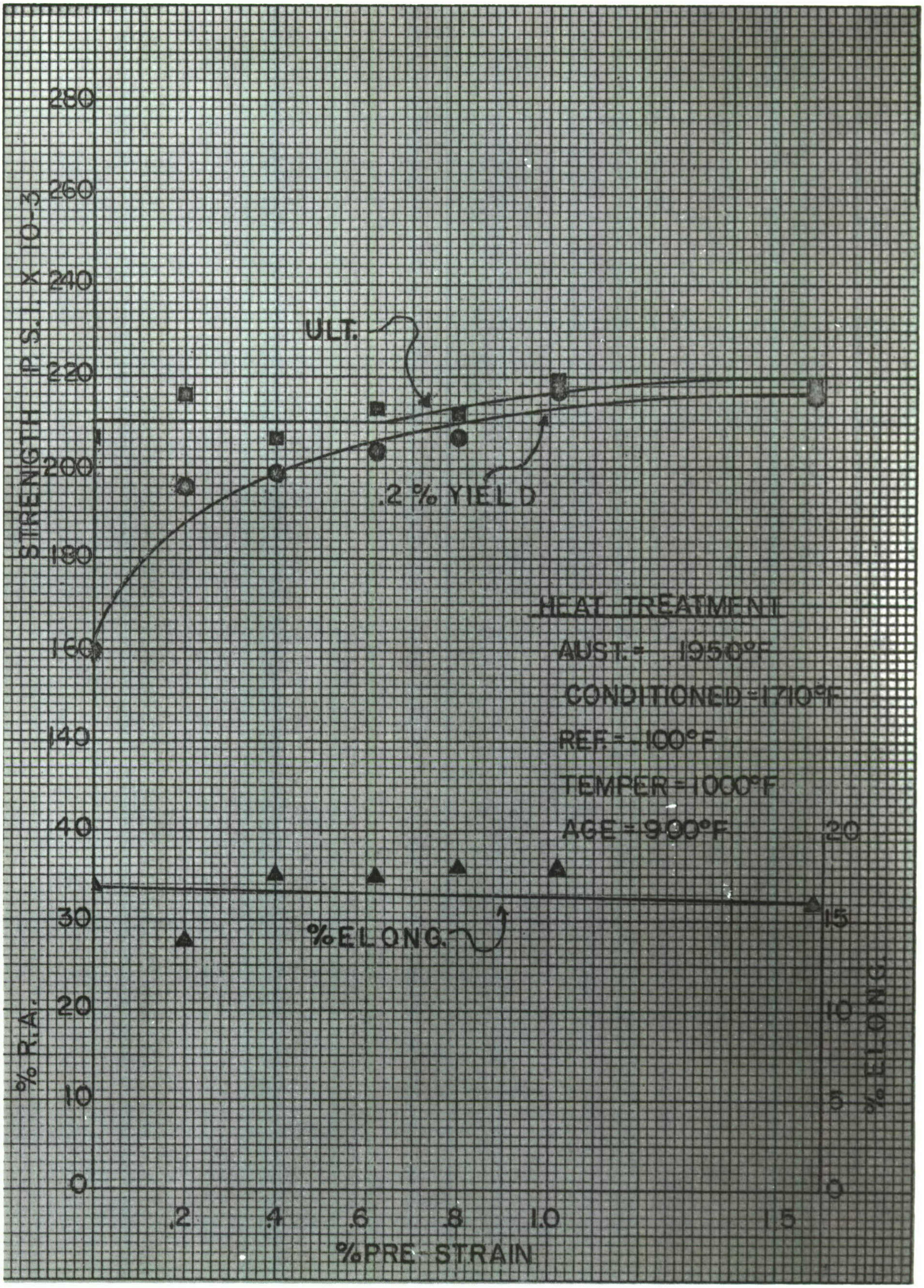



FIGURE 30

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF AM-355

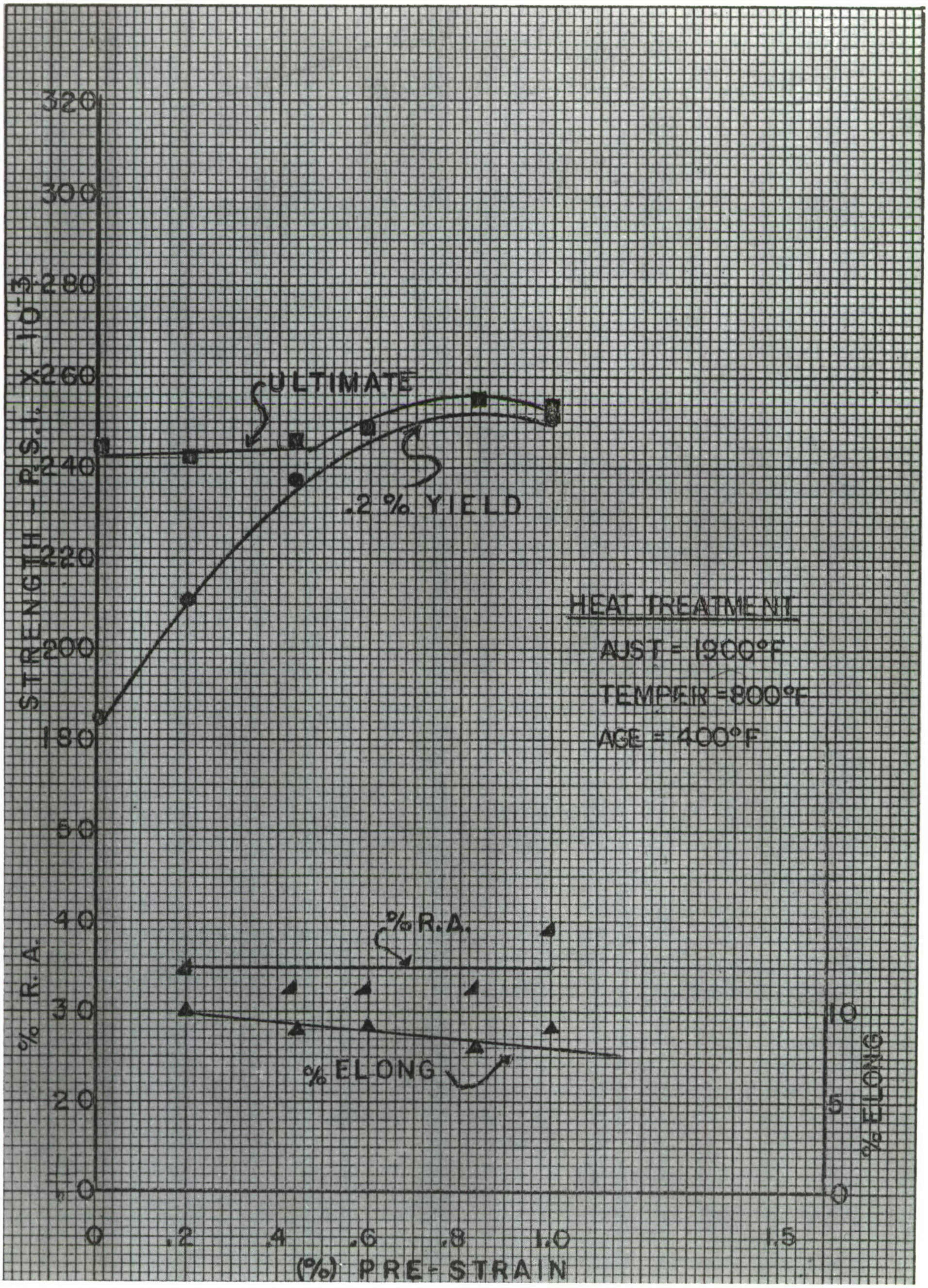



FIGURE 31

EFFECT OF PRE-STRAIN WITH NEARLY OPTIMUM AGING ON THE PROPERTIES OF 422

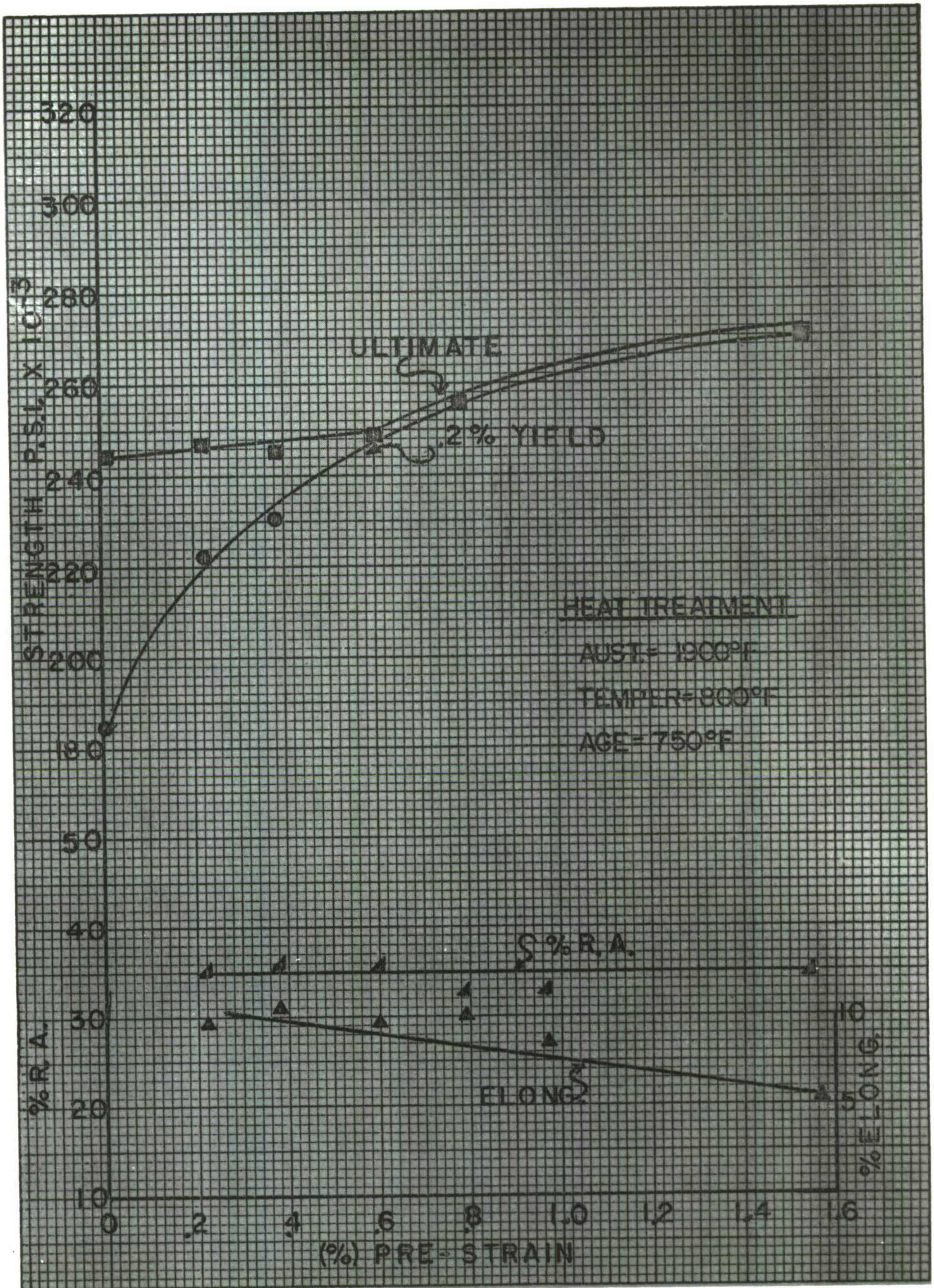



FIGURE 32

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF 422

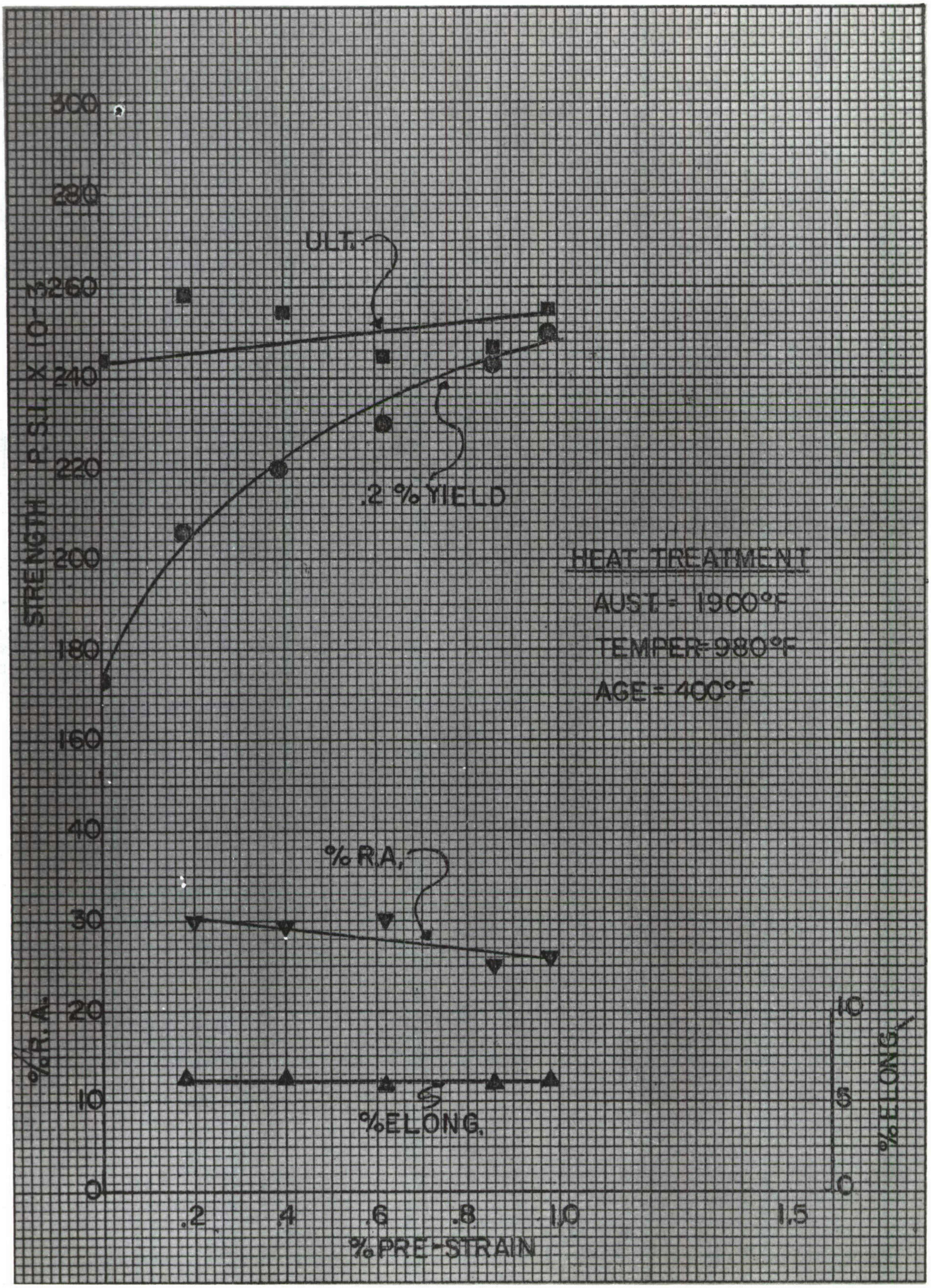



FIGURE 33

EFFECT OF PRE-STRAIN WITH LESS THAN OPTIMUM AGING ON THE PROPERTIES OF 422

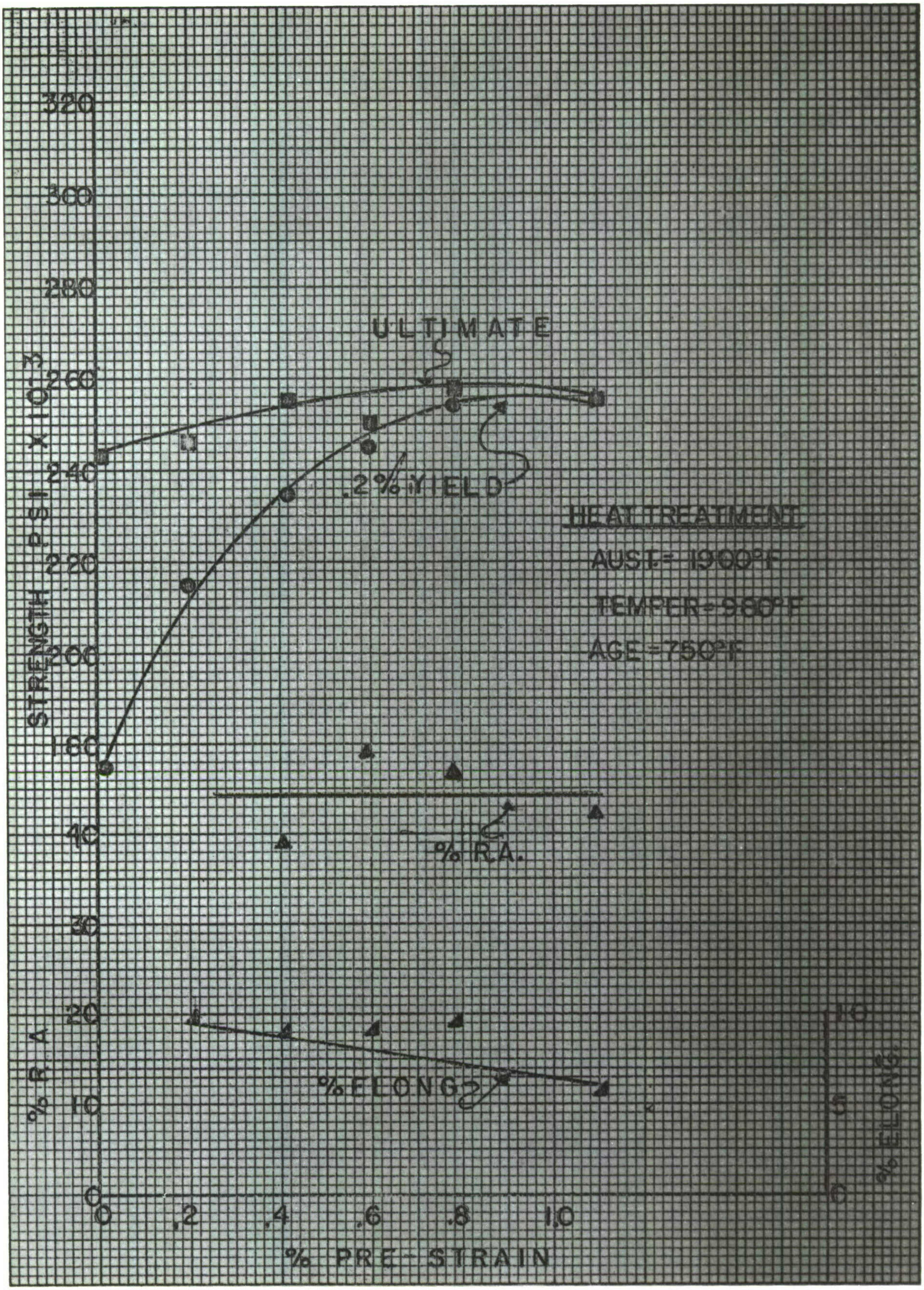



FIGURE 34

EFFECT OF PRE-STRAIN WITH OPTIMUM AGING ON THE PROPERTIES OF 422

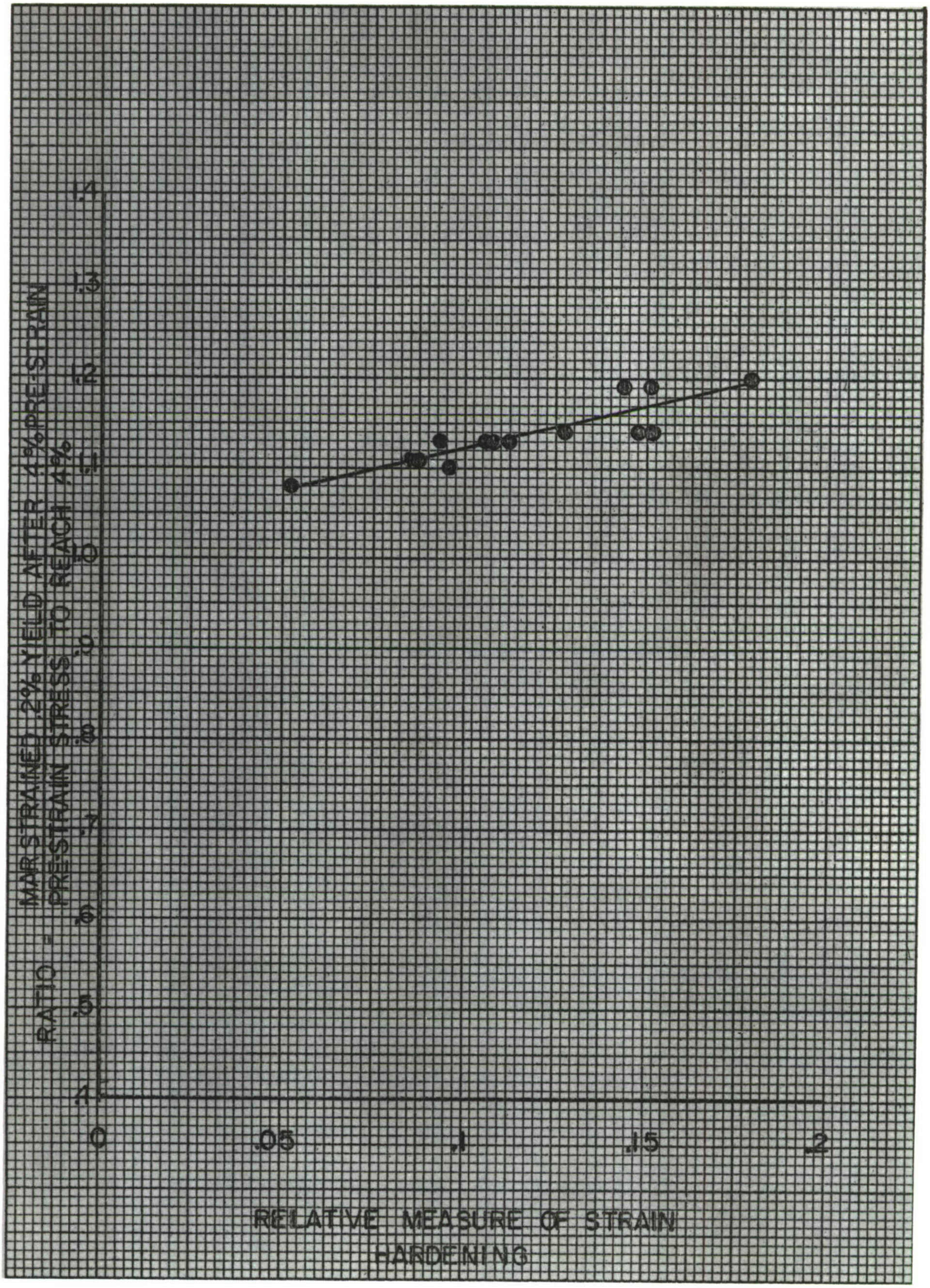



FIGURE 35
EFFECT OF STRAIN HARDENING ON MAR-STRAIN RESPONSE

## TABLE 3

## STRAIN HARDENING VS. MAR-STRAIN RESPONSE

| ALLOY | TEMPER | RATIO* | RELATIVE STRAIN HARDENING |
|---|---|---|---|
| 300M | 400°F | 1.14 | .152 |
| | 600°F | 1.13 | .106 |
| D6AC | 400°F | 1.13 | .112 |
| | 600°F | 1.13 | .108 |
| | 800°F | 1.08 | .052 |
| WCM-4 | 600°F (1) | 1.14 | .128 |
| | 600°F (2) | 1.19 | .144 |
| Mod. S-5 | 575°F | 1.13 | .092 |
| WHC | 600°F | 1.10 | .096 |
| Vascojet 1000 | 1000°F | 1.14 | .147 |
| AM-355 | 850°F | 1.11 | .087 |
| | 1000°F | 1.11 | .085 |
| 422 | 800°F | 1.19 | .151 |
| | 980°F | 1.20 | .179 |

*Ratio =

$$\frac{\text{Mar-Strained .2\% yield after .4\% pre-strain (optimum age)}}{\text{Pre-strain stress to reach .4\% strain}}$$

(1) Refrigerated
(2) Not refrigerated

strain hardening. If the stresses had been converted to true stress (using the true area vs. the original area) and the percent strain to true strain, this slope value would be the strain hardening exponent. The value used was in error by a few percent, but it showed the relationship of strain hardening to Mar-Strain response. As the strain hardening increases, the ratio of the two stresses increases. The values for each of the alloy conditions are shown in Table 3.

The data in Table 3 also show the effect of the tempered structure on the relative strain hardening of the alloy. One excellent example was the decrease in strain hardening as the tempering temperature of Ladish D6AC was increased. This decrease was caused by the loss of optimum carbide dispersion by the increasing tempering temperature. Secondary hardening which occurred with Vascojet 1000, 422SS, and AM-355, increased the strain hardening of these alloys at the higher temperature by more nearly optimum compound dispersion.

Specific results for each alloy were as follows:

300M - (Figures 17 and 18)

The 300M alloy can be Mar-Strained to over 280,000 psi .2% yield with .4% pre-strain when initially tempered at either 400 or 600$^{o}$F. A lower heat treated yield and ultimate (0% pre-strain) was exhibited by the 400$^{o}$F tempered material and was caused by less than optimum precipitation of epsilon carbide. The pre-strain and aging produced nearly identical properties for the two tempered conditions.

D6AC - (Figures 19, 20 and 21)

The response of D6AC differed from that of 300M. Higher properties were obtained for the 400$^{o}$F tempered material than the 600$^{o}$F. D6AC contained a

low amount of silicon compared to 300M and therefore the rate of decomposition of martensite was more rapid. Optimum dispersion of epsilon carbide was encountered at the lower 400°F temper which led to higher heat treated properties than was obtained for the 600°F temper. The Mar-Strain properties of 600°F tempered D6AC compared favorably to 300M tempered at 600°F - that is 280,000 psi, .2% yield was obtained after .4% pre-strain - because the faster rate of tempering of the D6AC alloy was counterbalanced by the higher carbon content (.47% carbon vs. 41% for 300M).

WCM-4 (Figures 22 and 23)

To obtain the maximum strength with this alloy (because of its carbon and nickel content), refrigeration to at least -100°F was required to effect complete transformation to martensite before tempering. Without refrigeration, a considerable amount of austenite was retained. Because this feature was available, it was decided to determine the effect of retained austenite on the Mar-Strain response.

The primary effect of the retained austenite was to reduce the strength level obtainable. The presence of the retained austenite did increase slightly the strain hardening of the alloy (.144 vs. .128) which increased the percentage increase in strength caused by aging, but this effect was not great enough to raise the response to the level obtainable by elimination of the retained austenite. This alloy, with refrigeration, can be Mar-Strained to over 300,000 psi .2% yield with as little as .4% pre-strain which is excellent for a .40% carbon steel.

Modified S-5 (Figure 24)

The heat of Modified S-5 which was evaluated demonstrated excellent response. Yield strength of over 300,000 psi was achieved with as little

as .2% pre-strain. This heat had .52% carbon which was on the upper end of the prescribed carbon level for the alloy. More pre-strain will be required for a lower carbon level of the alloy to achieve this strength level, but more as heat treated ductility will be available.

WHC (Figure 25)

The WHC alloy demonstrated good Mar-Strain response. Being capable of heat treatment to 280,000 psi yield strength and having good strain hardening characteristics, a yield strength of 320,000 psi was possible with only .4% pre-strain and 400°F aging.

Vascojet 1000 (Figures 26 and 27)

The strength data obtained from Mar-Straining Vascojet 1000 emphasized the importance of optimum aging and demonstrated the need for higher aging temperatures when an alloy has been tempered at high temperatures. Vascojet 1000, tempered at 1000°F; when aged at 600°F, required approximately .6% pre-strain to achieve a Mar-Strained yield strength of 275,000 psi, whereas when aged at 800°F, only .3 to .4% pre-strain was required.

AM-355 (Figures 28, 29 and 30)

The importance of optimum aging was again demonstrated with AM-355. The 700°F age for AM-355 tempered at 850°F gave much higher Mar-Strain properties than was achieved with a 400°F age. A yield strength of the order ot 220,000 psi was achieved by Mar-Straining AM-355 after tempering at 850°F. A Mar-Strained yield of 200,000 psi was obtained for the 1000°F temper.

422 Stainless (Figures 31, 32, 33 and 34)

This alloy also demonstrated excellent response to Mar-Straining. This response was possible because of the excellent strain hardening

characteristics and good aging response. Two aging temperatures, 400 and 750°F, were used for the two heat treatments, 800 and 980°F tempers. The 800°F temper responded equally well to both aging conditions, but the 980°F temper required the 750°F age to achieve good response. Both tempers can be Mar-Strained to over 230,000 psi yield strength with as little as .4% pre-strain when optimumly aged.

In summary, the magnitude of the increase in strength obtained by Mar-Straining was dependent on the heat treated strength, the strain hardening characteristics and the aging response.

## D.) Summary and Conclusions

From the study of four classes of steels, low alloy, secondary hardening, semi-austenitic and martensitic stainless, several conclusions which are believed to be applicable to the adaptation of the Mar-Strain process to other alloy systems have been obtained. They are pertinent to optimization of the process using small amounts of pre-strain (approximately .4%) and short aging times - two hours maximum. At greater amounts of pre-strain and longer aging times, slightly different conditions may prevail to obtain optimization. The conditions studied, .4% pre-strain and short aging times, are advantageous for the application of the Mar-Strain process to solid propellant rocket motor cases.

1) Alloys which have been tempered to produce precipitated metastable compounds of carbon or nitrogen (e.g., epsilon carbide) should be aged (two hour maximum) in the temperature range of 300 to 400°F.
2) Alloys which have been tempered to precipitate more stable and/or complex compounds of carbon (e.g. $Fe_3C$) and nitrogen

($Fe_4N$) must be aged in the temperature range of from 600 to 800$^{o}$F to achieve optimum aging.

3) Using optimum aging, pre-strain of the order of .4 to .5% can be applied without affecting the ultimate strength of the alloy.

4) Ductility as measured by percent reduction in area and elongation were not appreciably affected by pre-strains of .4% or less.

5) The magnitude of the yield strength increase obtainable by Mar-Straining each alloy was dependent on the value of the strain hardening exponent. The larger the strain hardening exponent, the larger was the increase in strength.

## E.) Alloy Selection For Phase II

Four criteria were used as a basis for the selection of two alloys for determination of their engineering properties.

1) Mar-Strained strength capabilities
2) As heat treated notch toughness
3) Commercial availability
4) Utilization of the alloy in present or future weapon systems

The steels selected were to be representative of two different Mar-Strained strength levels, 275,000 and 300,000 psi .2% yield. Three of the compositions investigated produced Mar-Strained yield strength equal to or in excess of 300,000 yield. These were G.E. development alloys, WCM-4 and WHC, and a G.E. modification of a S-5 tool steel. Three alloys also demonstrated the capability of obtaining a 275,000 psi yield strength - 300M, D6AC, and Vascojet 1000.

The importance of the second criteria, notch toughness, cannot be overemphasized. Pressure vessel performance and reliability are directly related to the toughness of the alloy and the manufacturing skill and inspection procedures used. The greater the notch toughness of the alloy, the greater is the tolerance to defects introduced during the manufacturing operations. Low notch toughness requires stringent inspection procedures to assure removal of defects in order to insure reliability in proof testing and subsequent use in missile systems. The Mar-Strain process, since it requires the vessel to be plastically strained, requires good notch toughness and inspection procedures. Laboratory notch testing of alloys has been found to be useful in rating alloys with regard to increasing or decreasing order of toughness. Toughness comparisons must be made at

nearly equal yield strengths, thicknesses and testing temperatures because these factors have a pronounced influence on the notch behavior of any alloy composition.

Unpublished laboratory data as well as data in the literature have served to establish good quantitative data for notch comparison of the alloys which have met the strength requirements of this contract. The as heat treated notch ductility of the three compositions capable of achieving the 300,000 psi Mar-Strained yield strength can be rated in the following decreasing order of toughness.

1) WCM-4 - 250,000 psi yield

2) Modified S-5 - 275,000 psi yield

3) WHC - 275,000 psi yield

The alloys capable of achieving a 275,000 psi Mar-Strained yield strength level can also be rated in descending order of toughness:

1) 300M - 240,000 psi yield

2) D6AC - 240,000 psi yield

3) Vascojet 1000 - 240,000 psi yield

The third and fourth criteria, although separated above, are closely related. Alloys presently being utilized in Missile and Weapon systems are those which are most commercially available. The three alloys capable of the lower Mar-Strained strength level, 300M, D6AC and Vascojet 1000 have been or are being highly utilized by industry. The D6AC composition is presently the alloy which is being utilized to the greatest extent by the missile industry. The three alloys meeting the 300,000 psi yield strength level are at present essentially development alloys. The S-5 tool steel has been used in the machine tool industry for several years, and the

G. E. modified version is presently being applied to development rocket case hardware. A ten ton commercial vacuum arc remelted heat of the modified chemistry has been produced. The other two G.E. development compositions have only been produced in heats up to 50-500 pounds for metallurgical evaluation purposes.

In conjunction with the ASD project engineer, D6AC and Modified S-5 were chosen as the alloys for determination of their engineering properties based on the best compromises of the above criteria. Although the engineering property data generated would be more directly applicable to the alloys studied, D6AC and Modified S-5, the effects of the Mar-Strain process on these properties should be indicative of the effects which would result if any of the other alloys were Mar-Strained, e.g. if Mar-Straining increased the fatigue properties of D6AC, similar increases would be expected if the process were applied to 300M or Vascojet 1000.

## VI. PHASE II

## ENGINEERING PROPERTIES

### A.) Introduction

The engineering properties determined can be categorized into two classes, uniaxial and biaxial. The uniaxial stresses were applied by tension and the biaxial by internal pressure. The biaxial evaluation was confined to a 2 to 1 stress field as is encountered in the wall of a cylindrical pressure vessel with a length to diameter ratio greater than 1.

Included in the uniaxial evaluation were tensile properties, fatigue properties and notch properties. The fatigue properties included both the high cycle-low stress and low cycle-high stress conditions. Notch properties were ascertained using an internally notched specimen as specified by the ASTM Committee on Fracture Testing of High Strength Sheet Materials. These uniaxial properties were determined on .050 inch thick Modified S-5 and on .080 inch Ladish D6AC.

Six inch diameter cylinders were used to determine the biaxial properties. Wall thicknesses were the same as the sheet thicknesses used in the uniaxial evaluations. The properties obtained included circumferential (hoop) yield strength, burst strength, and low cycle fatigue resistance.

### B.) Material

Sheet and ring forgings of each alloy were procured from the same vacuum arc remelted heat for determination of the engineering properties. Complete chemical analyses were obtained on the sheet and carbon analysis was obtained from one of the ring forgings. The results are shown in the following tabulation.

## CHEMICAL ANALYSIS

### Modified S-5

| | C | Mn | Si | Ni | Cr | V | Mo |
|---|---|---|---|---|---|---|---|
| Nominal | .49 | .80 | 1.8 | - | - | .25 | .50 |
| Sheet | .455 | .64 | 1.62 | - | - | .26 | .52 |
| Cylinder | .48 | | | | | | |

### Ladish D6AC

| | C | Mn | Si | Ni | Cr | V | Mo |
|---|---|---|---|---|---|---|---|
| Nominal | .46 | .75 | .20 | .60 | 1.0 | .1 | 1.0 |
| Sheet | .469 | .67 | .22 | .46 | 1.20 | .05 | 1.20 |
| Cylinder | .47 | | | | | | |

All of the analyses were within normal tolerances for vacuum arc remelted steels with the exception of the carbon analysis of the Modified S-5 sheet and the vanadium content of the D6AC.

As rolled sheet thicknesses were .125 for D6AC and .110 for Modified S-5. The ring forgings were nominally 1/2 inch in wall thickness, 5 3/4 inch I.D. and 18 inches long. All material was received in the spheroidize annealed condition except the Modified S-5 sheet which was received in the mill annealed condition.

## C.) Tensile Testing

### Tensile Properties of Modified S-5

The sheet was sheared into 1.6 X 6 inch strips and spheroidize annealed (1450$^{\circ}$F-1/2 hour, air cooled to room temperature, re-heated to 1350$^{\circ}$F and held 8 hours). The 6 inch direction of the strips was cut transverse to the final rolling direction. The hardness after the spheroidize

was Rb 88-90. Standard ASTM sheet tensile specimens were machined (.500 gage width - 2 inch gage length). The specimens were coated with a "No-carb" coating and austenitized at 1650°F in an argon atmosphere. The specimens were quenched in oil, refrigerated to -100°F in a mixture of dry ice and alcohol and then tempered for 2 + 2 hours at 600°F. This heat treat procedure differed from the heat treatment used in determining the Mar-Strain response of this alloy. The change in heat treatment was introduced to assure a heat treated yield strength of 275,000 psi. The change was required because of the lower than desired carbon content of the acquired sheet material. The change was made based on a study of similar chemistry sheet material. If the original heat treatment were used (1650°F austenitize, salt quench and a 575-2+2 hour temper), this .45 carbon material could only achieve a 265,000 psi yield. After heat treatment, the specimens were surface ground to .050 inches in thickness.

To determine the Mar-Strain tensile properties, three specimens were pre-strained at each of the following nominal amounts of strain, .2, .4, .6, .8 and 1.0%. The specimens were aged for two hours at 400°F. The specimens were then tested (.005 inches/inch/min. strain rate) and the tensile properties obtained. The dimensions of the specimens after pre-strain and aging were used to calculate the tensile properties.

The tensile properties obtained are shown in Figures 36 and 37. The ultimate strength, yield strength, elongation and reduction in area are plotted versus percent pre-strain. The ultimate stresses fell reasonably within a 16,000 psi scatter band which corresponded to the 16,000 psi variation encountered in the as heat treated (0% pre-strain) S-5 material. The scatter in yield was not nearly as consistent as the ultimate. The

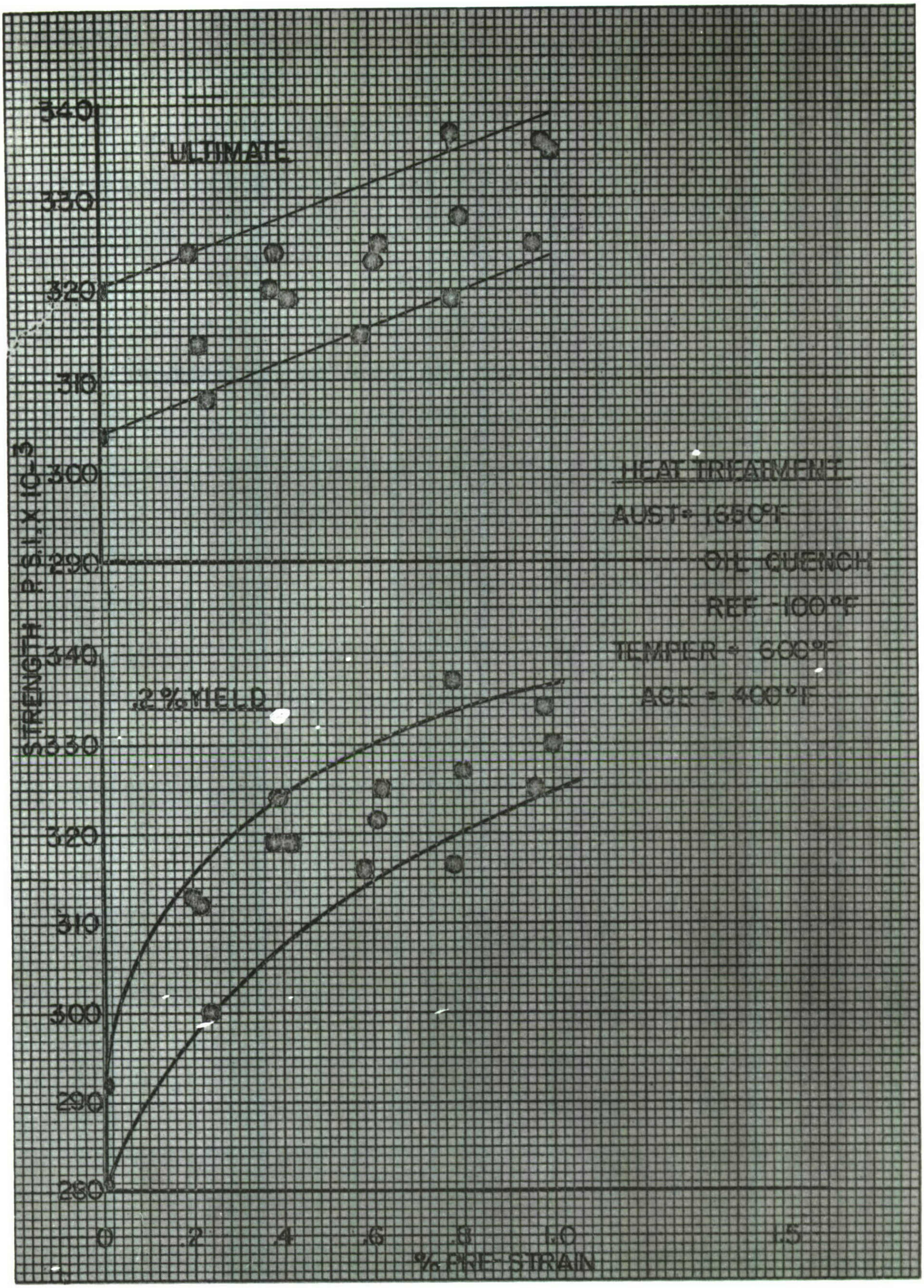



FIGURE 36

TENSILE PROPERTIES OF MAR-STRAINED MODIFIED S-5

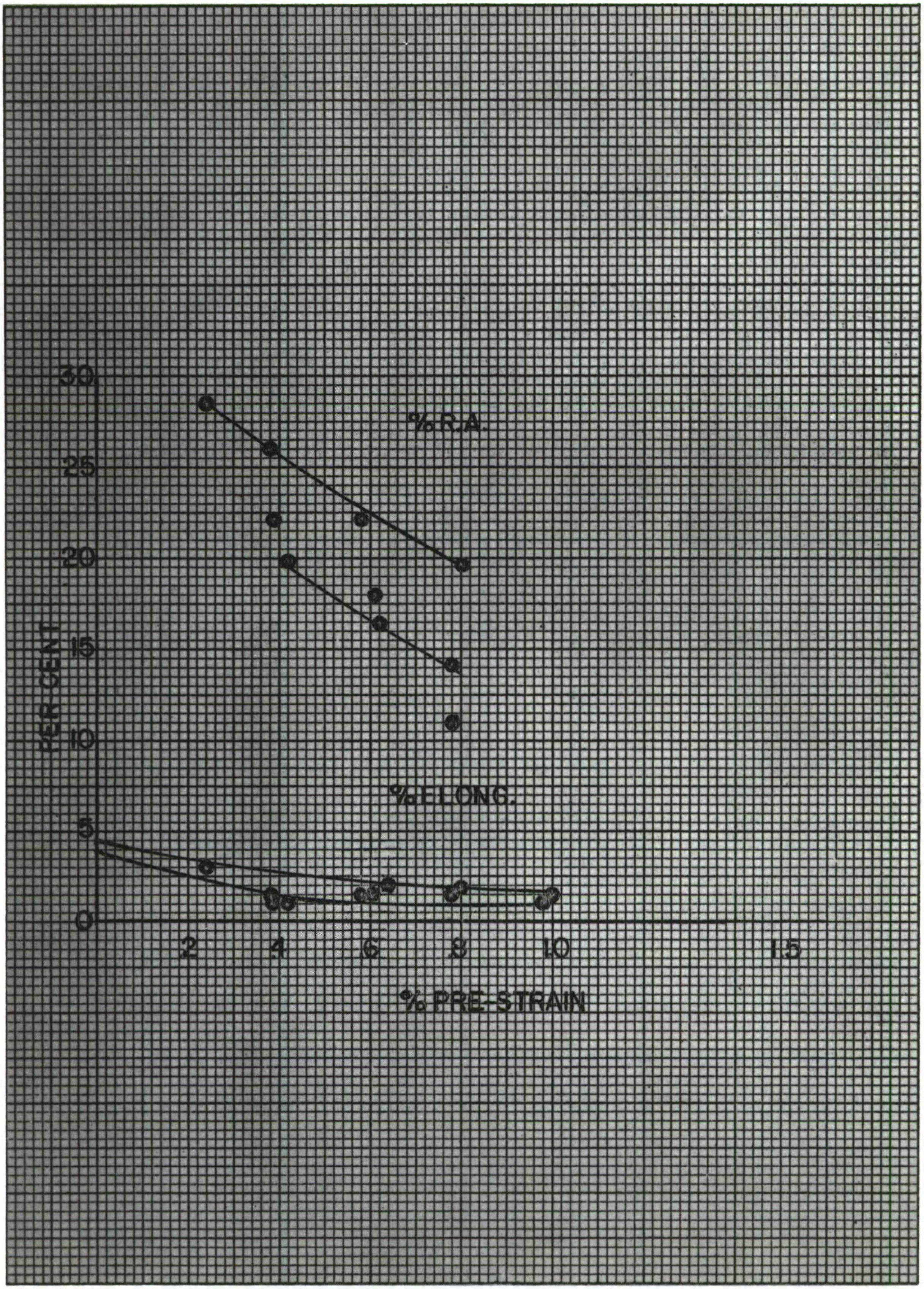



FIGURE 37
TENSILE PROPERTIES OF MAR STRAINED
MODIFIED S-5

scatter in yield strength varied from 12,000 to 16,000 psi. Ductility, as measured by reduction in area, dropped steadily with percent pre-strain. Elongation also decreased with increased pre-strain up to .4% and then held constant. This drop in elongation, with small amounts of pre-strain, differed from that which was experienced in the Mar-Straining of other alloys - where the elongation was not affected by pre-strains of this magnitude.

In order to determine the consistency of the aging response, the ratio of the new .2% yield strength (Mar-Strained .2% yield) and the stress encountered during pre-straining was calculated and plotted vs. the percent pre-strain. The values obtained are plotted in Figure 38A. The ratios varied from approximately 1.08 to 1.10 which demonstrated a reasonably consistent aging response. This aging response was not appreciably affected by the amount of pre-strain (a very slight decrease was noted as the pre-strain increased). The ratios encountered were lower than were experienced in the Mar-Strain response study of this alloy. A value of 1.13 was previously obtained with a relative strain hardening value of .092. A relative strain hardening value of only .062 was obtained with this .45% carbon heat which predicts a ratio of 1.09 (Figure 35), a value which lies mid-point in the band of ratios obtained. The difference in strain hardening can be identified with the change in chemistry and in heat treatment required to achieve the desired heat treated yield strength.

The ratio of the Mar-Strained 2% yield strength and the .2% yield encountered during the pre-straining was also calculated for each specimen and plotted versus the percent pre-strain (Figure 38B). This figure can be used as a yield strength prediction curve. With knowledge of the heat

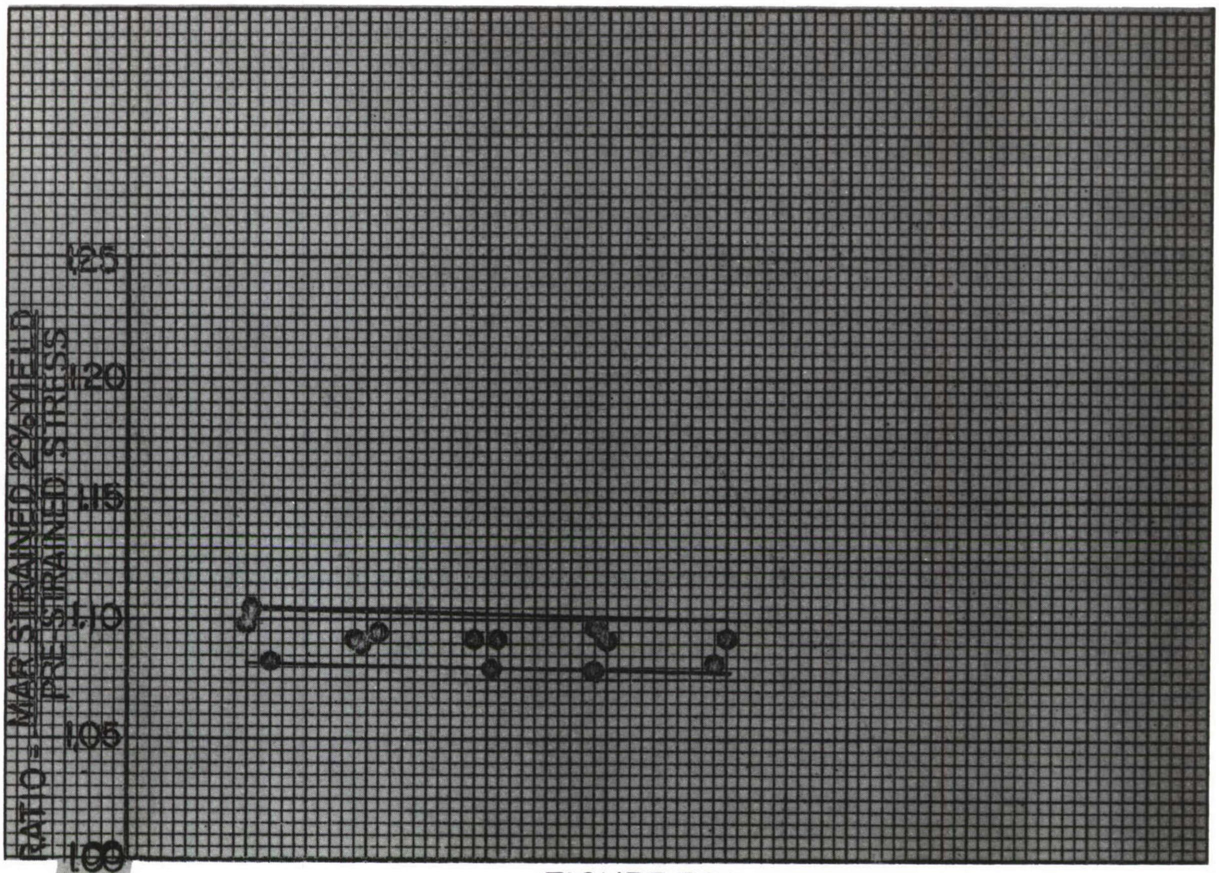



FIGURE 38A
AGING RESPONSE OF MOD. S-5

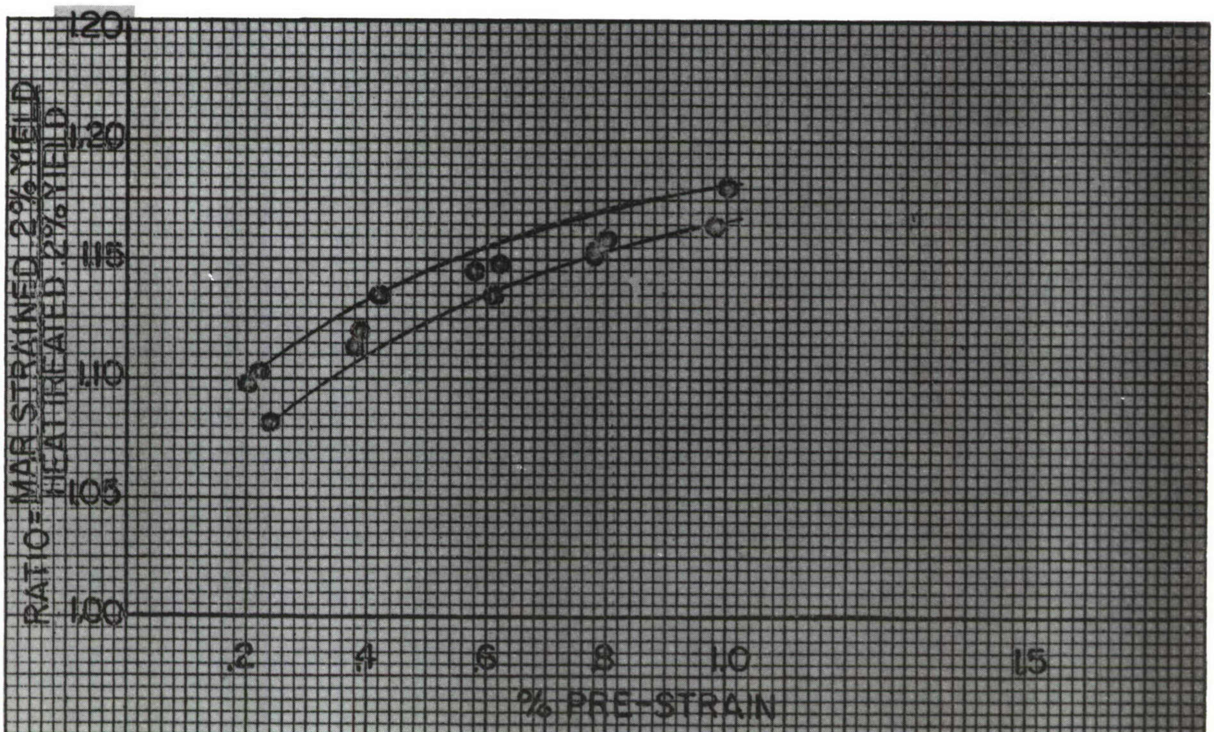



FIGURE 38B
YIELD STRENGTH PREDICTION OF
MAR STRAINED MOD. S-5

treated yield strength, a minimum Mar-Strain yield strength can be predicted for any amount of pre-strain (provided the relative strain hardening is not varied by heat treatment or chemistry changes). This curve can be used in other property evaluations where actual measurement of the Mar-Strained yield strength is not possible. These values were also found to lie in a fairly narrow band of .02 in width.

The data have shown that the aging response, the as heat treated strength and the strain hardening characteristics all affect the Mar-Strain yield strength with the heat treated strength producing the major amount of spread in the tensile properties. To achieve the desired Mar-Strain yield strength of 300,000 psi, a minimum of .25% pre-strain (with a heat treated yield strength of 280,000 psi) was required.

Tensile Properties of Mar-Strained Ladish D6AC

Tensile specimens were prepared in the same manner as with the Modified S-5. The specimens were austenitized at 1650$^{o}$F in a carbon potential furnace with a dew point controller on the exit gas. The dew point was set to give a carbon potential of .46% carbon for D6AC material. The specimens were quenched in salt at 400$^{o}$F for five minutes, cooled to room temperature, and then tempered for 2 + 2 hours at 600$^{o}$F. The specimens were then ground to .080 inches in thickness, equal amounts being removed from each side to remove all traces of possible decarburization. The heat treatment selected differed from the one originally evaluated (Table 2) for Mar-Strain response. The austenitizing condition was changed from a two cycle operation to a one cycle. This change was made to simplify the heat treatment to be used with the cylinders.

Three specimens were pre-strained at each of the following percentages, .2, .4, .6, .8, and 1.0. The specimens were then aged at 350°F (Maximum aging could be obtained between 300°F and 400°)for two hours and tensile tested. The dimensions of the specimens after pre-strain and aging were used to calculate the tensile properties.

The Mar-Strained tensile properties obtained are shown in Figures 39 and 40. The ultimate strength curve was more indicative of the type of curve previously obtained with Mar-Strained alloys. The ultimate was not affected by pre-strain up to .4%. At this point, the ultimate increased with pre-strain. The spread in ultimate was low, 6 to 10,000 psi- about what was experienced with heat treated material. The yield strength also gave a narrow band spread in properties, very nearly equal to the spread found in the heat treated condition. Unfortunately, the heat treated strength level produced with this heat treatment was lower than desired. A normally expected yield strength for D6AC with a 600°F temper is between 230,000 and 240,000 psi. This heat treatment produced a maximum yield strength of 230,000 psi.

The aging response of this heat treatment was excellent (Figure 41A). A relative strain hardening value of .096 was calculated which predicts a ratio of 1.115 for the Mar-Strained .2% yield over the pre-strain stress. The actual ratios calculated varied from 1.10 to 1.125 -excellent agreement with the predicted values.

The yield strength prediction curve (Figure 41B) also demonstrated the excellent Mar-Strain response of this alloy. A very narrow spread occurred. The curve predicts a minimum yield strength for .4% pre-strain of only 265,000 psi (225,000 minimum heat treated yield strength). This is 10,000

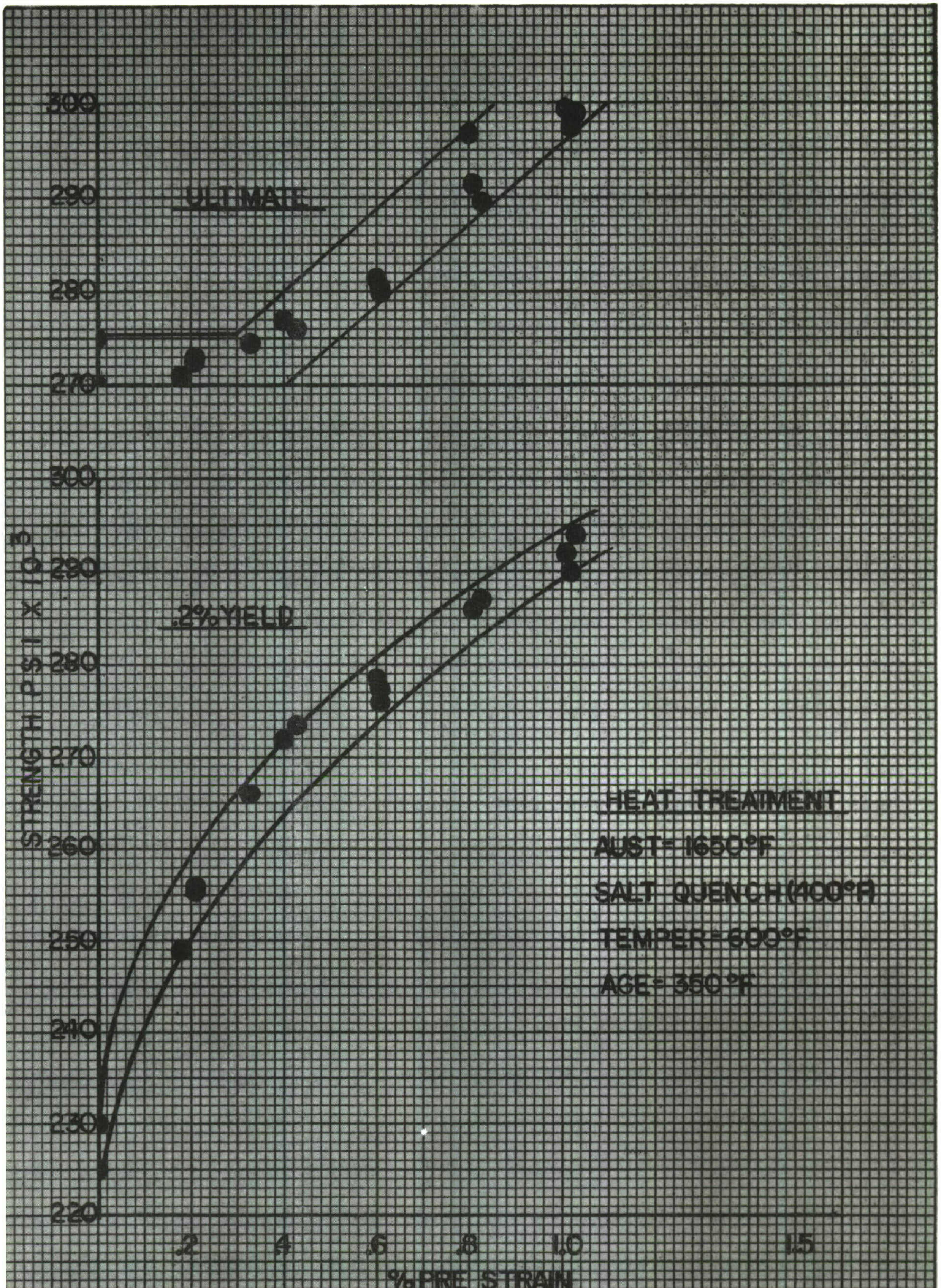



FIGURE 39
TENSILE PROPERTIES OF MAR-STRAINED
D6AC

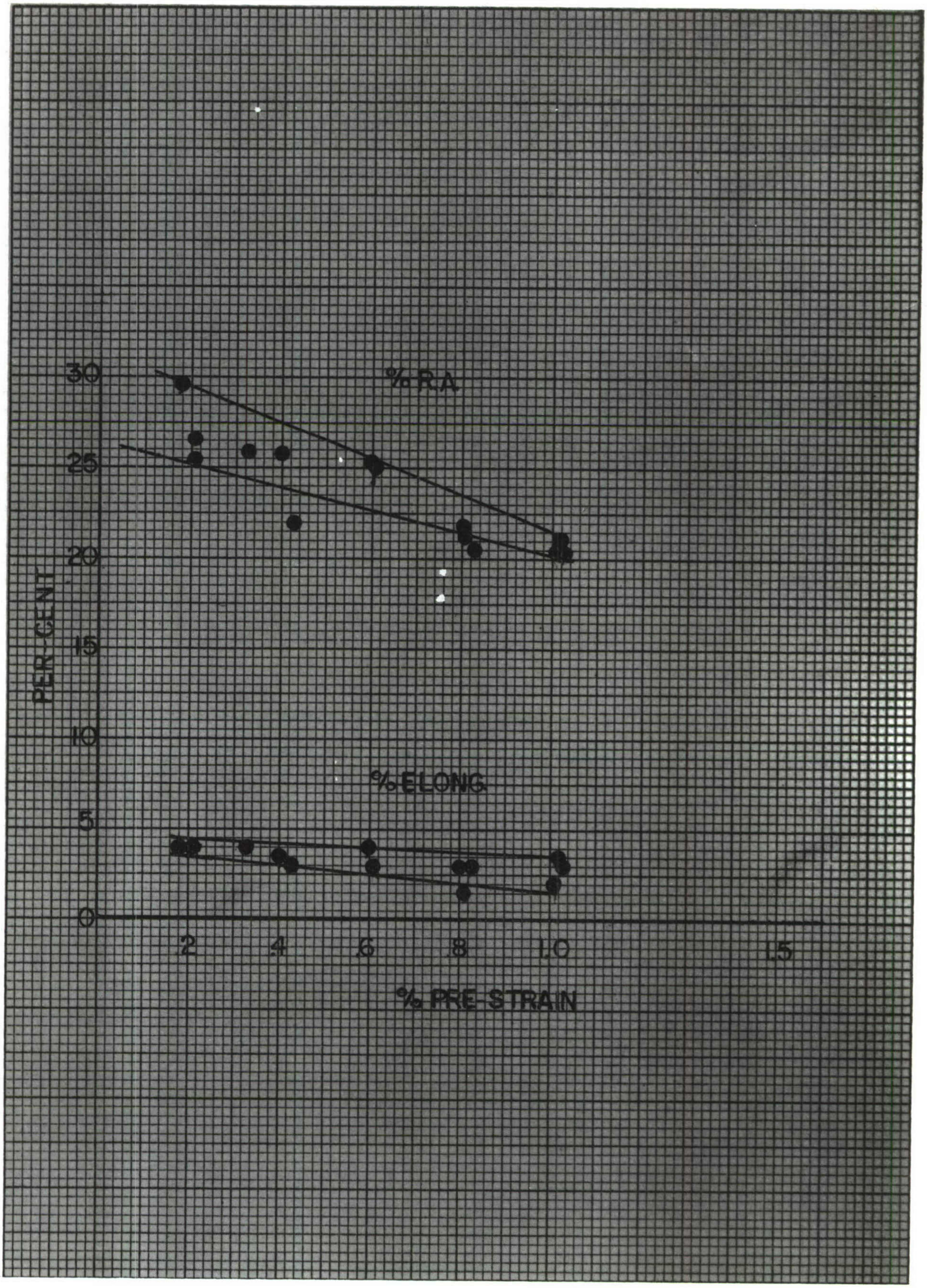



FIGURE 40
TENSILE PROPERTIES OF MAR STRAINED
D6AC

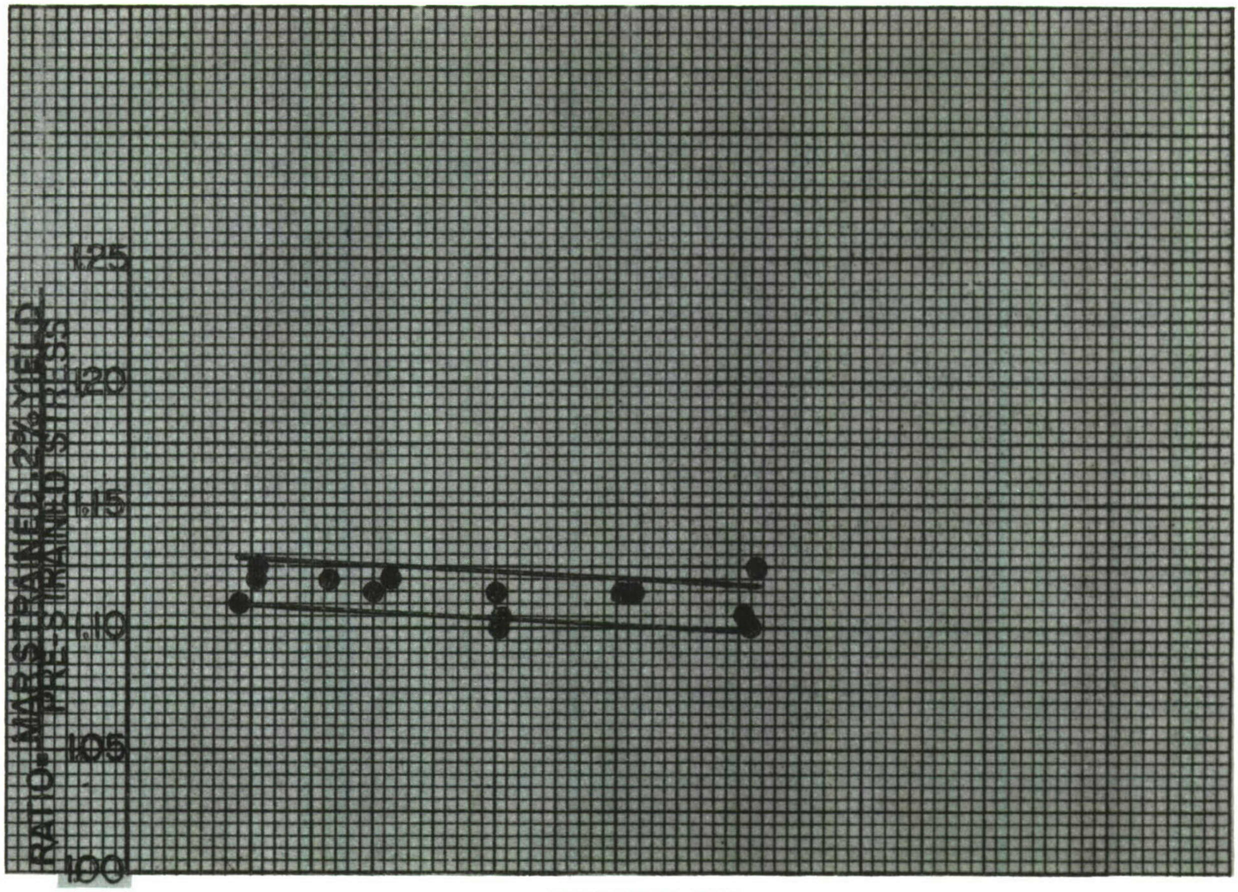



FIGURE 41A

AGING RESPONSE OF D6AC

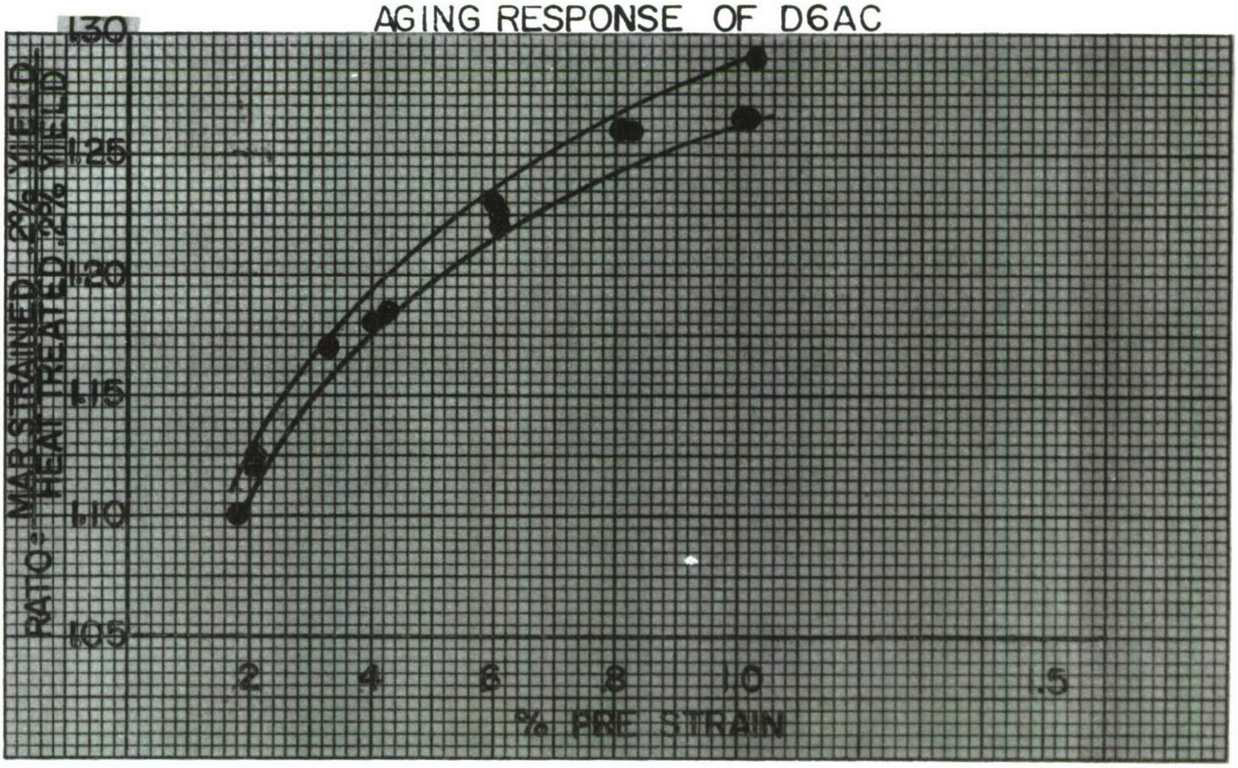



FIGURE 41B

YIELD STRENGTH PREDICTION OF D6AC

psi lower than the desired 275,000 psi because of low initial yield strength.

Since the chemical analysis of the sheet showed that the carbon level was acceptable for D6AC, the heat treatment used was suspected to be the reason for the low yield strength obtained. Several other heat treat cycles were then investigated in an attempt to improve the heat treat response. The results obtained with these cycles ,as well as the average results obtained with the original cycle,are shown in Table 4. Significant yield strength increases were obtained with three of the four cycles investigated. Since the heat treat cycle selected for uniaxial studies was also to be used for biaxial studies, the choice of cycle was governed by the facilities available for cylinder heat treatment. The number 5 cycle (1650$^{o}$F Normalize, 1550$^{o}$F Austenitize, Oil Quench) was preferred primarily because of equipment limitations.

The Mar-Strain tensile properties using the new cycle were obtained, but were only secured up to .6% pre-strain. The results obtained are shown in Figure 42 through 44. The results were similar to those obtained with the original heat treatment with the exception that the minimum yield strength curve had been raised 10,000 to 12,000 psi at all levels of pre-strain. Aging response and yield strength prediction curves were similar, indicating that the strain hardening characteristics of the two cycles were nearly identical. With the new cycle, the desired 275,000 psi yield strength could be obtained with as little as .37% pre-strain.

TABLE 4

Average D6AC Tensile Properties for Various Heat Treat Cycles

| Cycle* | Ultimate Psi | .2% Yield Psi | Elong. % |
|---|---|---|---|
| 1) 1650°F Aust.** <br> 400°F Salt Quench | 273,000 | 227,000 | 4.5 |
| 2) 1650°Aust. <br> 400° F Salt Quench <br> -100°F Ref. | 284,000 | 237,200 | 4.7 |
| 3) 1650°F Aust. <br> Oil Quench | 273,700 | 231,500 | 4.5 |
| 4) 1650°F Normalize <br> 1550°F Aust. <br> 400°F Salt Quench | 276,000 | 238,500 | 4.5 |
| 5) 1650°F Normalize <br> 1550°F Aust. <br> Oil Quench | 274,700 | 237,200 | 4.7 |

*600°F - 2 + 2 hr. temper used for all cycles.

**Original heat treatment evaluated.

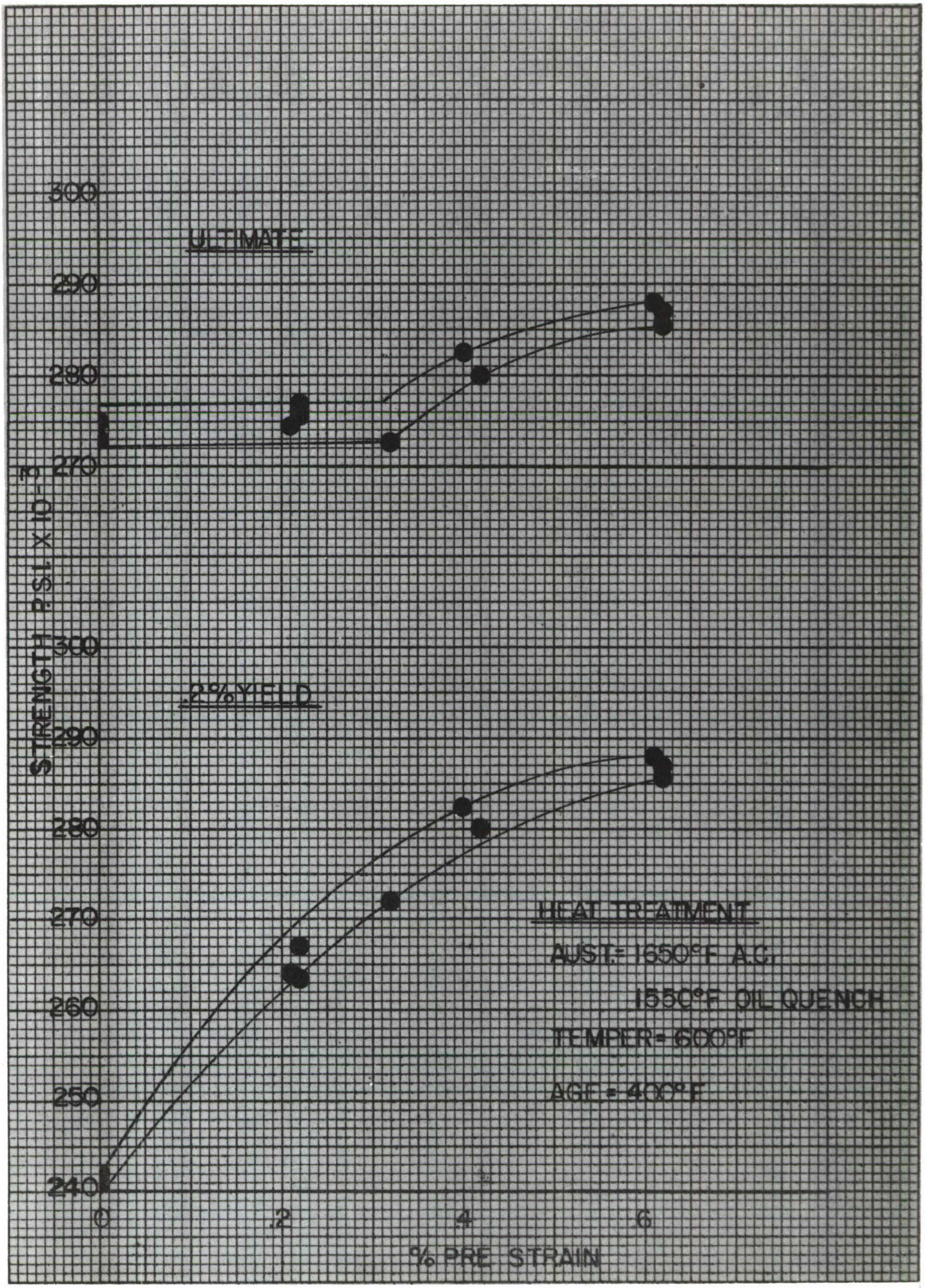



FIGURE 42
TENSILE PROPERTIES OF MAR STRAINED
D6AC

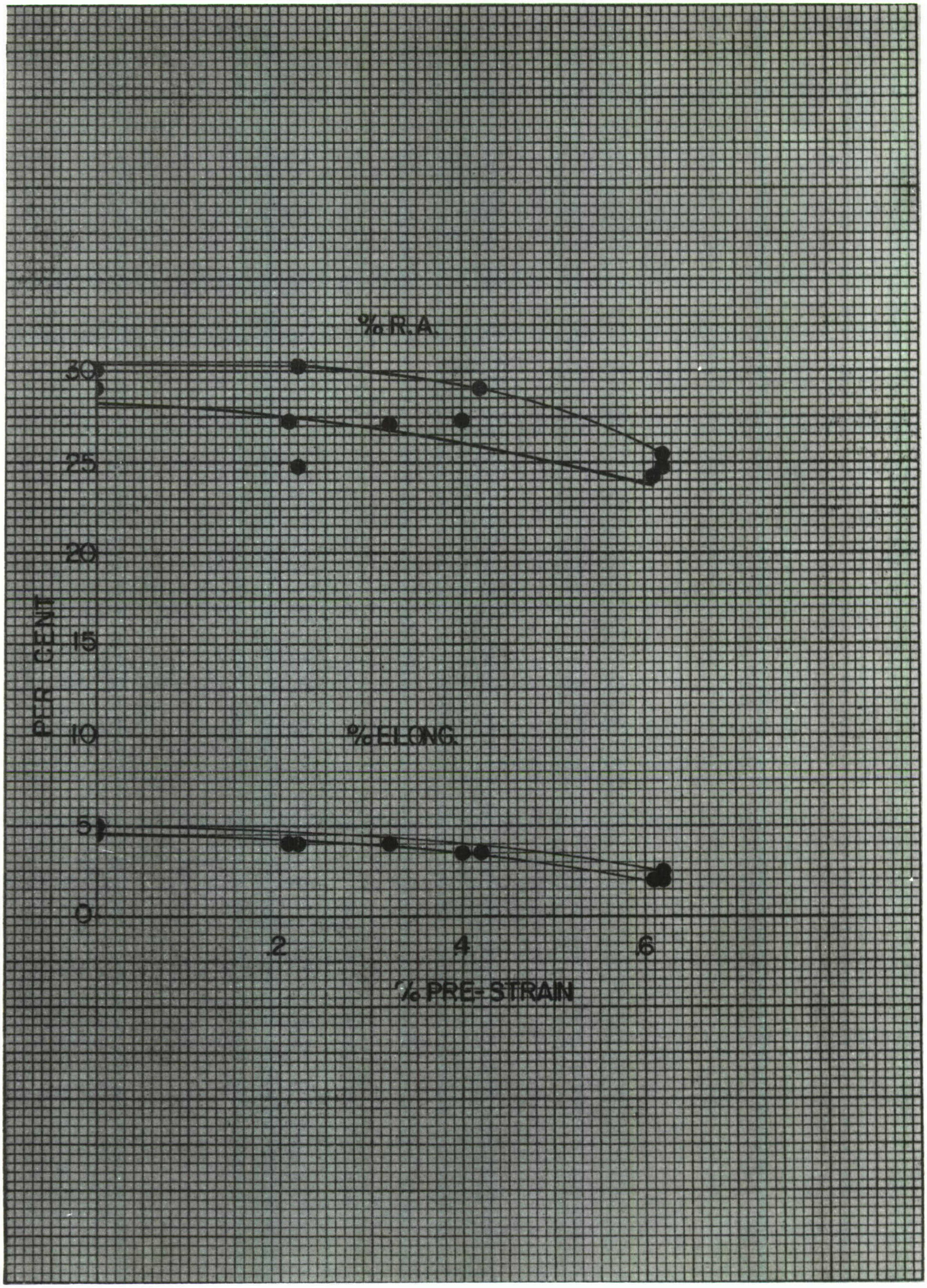



FIGURE 43

TENSILE PROPERTIES OF MAR-STRAINED D6AC

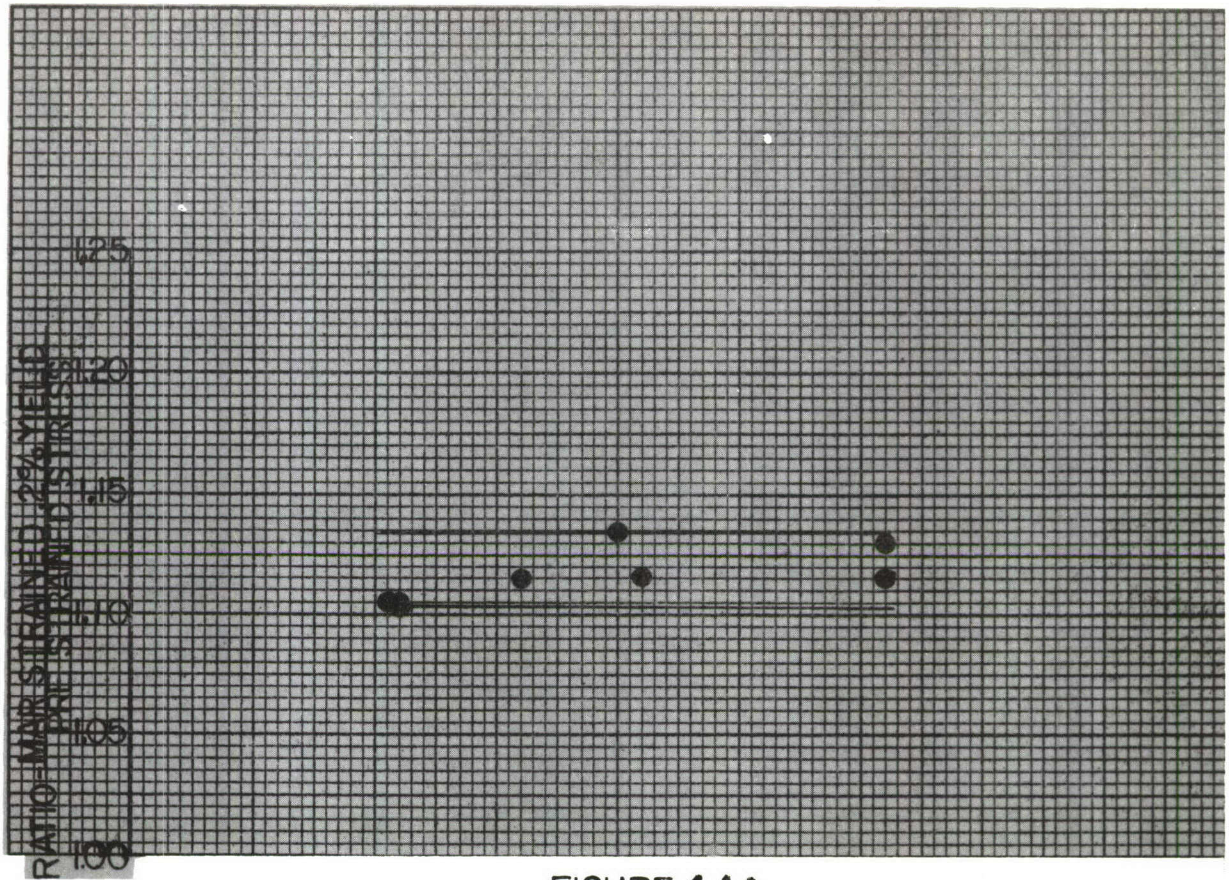



FIGURE 44A

AGING RESPONSE OF D6AC

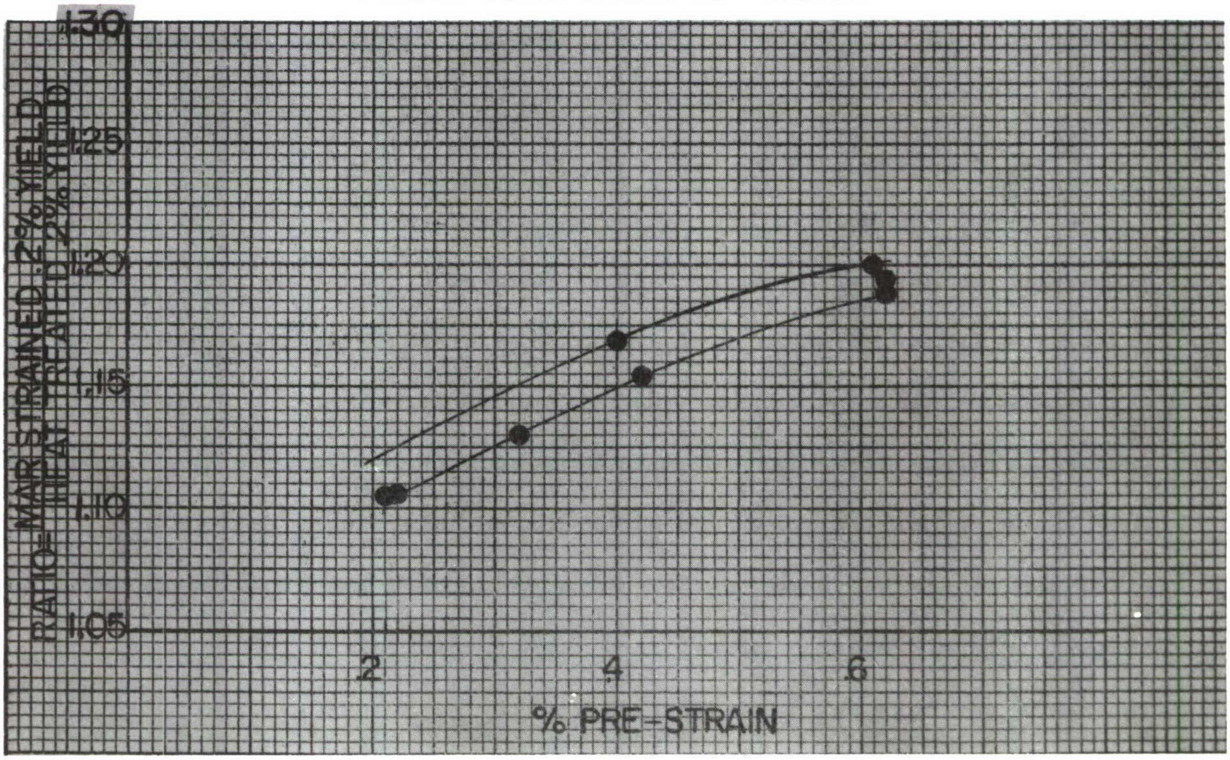



FIGURE 44B

YIELD STRENGTH PREDICTION OF

D6AC

## D.) Center Notch Properties of Mar-Strained Alloys

### Background

Two notch toughness measurements are significant to the application of the Mar-Strain process to production hardware. These are the notch toughness of the alloy in the heat treated condition and the notch toughness of the alloy after Mar-Straining. Good notch toughness is required in order to apply plastic strain to the hardware. Marginal toughness can be tolerated if good design and inspection techniques are used. The toughness after Mar-Straining becomes important if damage to a part is anticipated during further fabrication or installation in a weapon system. A third area of toughness is of interest, but is difficult to measure by present methods. This is the effect of straining and aging on defects in the heat treated structure which were subcritical for causing failure during the initial straining. The first two toughness values can be measured by laboratory tests and quantitative data obtained. The third toughness area of interest can only be inferred.

Recently, several papers have appeared in the literature which have described the effects of pre-stress (including stresses past the proportional limit) on the notch strength of high strength steels. These pre-stresses have been applied both before and after notching. Steigerwald (3) investigated the effect of warm pre-stressing (pre-stressing above the full shear transition temperature) on the notch strength of an H-11 steel and found that warm pre-stressing of notched specimens prior to testing to the breaking load at room temperature raised the notch strength. The notch strength was raised proportionally with increased pre-stress.

This increase in room temperature notch strength was attributed to the introduction of compressive residual stresses at the notch front by the pre-stressing. Although not discussed by Steigerwald, the temperature used for warm pre-stressing, 600°F, would bring about aging in the plastic strained area in the notch front which could have had some beneficial effect. Steigerwald also presented the effect of pre-stress prior to introduction of the notch on the room temperature notch strength. A decrease in notch strength occurred when the pre-stress was greater than the proportional limit. This decrease was noticed regardless of whether the pre-stressing was done at room temperature or at 600°F.

Srawley and Beachem (4) have also shown that warm pre-stressing of notched specimens above the ductile to brittle transition temperature increases the room temperature notch strength. They proposed that the increase was caused by blunting of the notch by plastic strain during the pre-stressing.

This information suggested that pre-straining would have a beneficial effect on notches present before pre-straining (as long as they are smaller than critical size for failure at the pre-strain stress) and aging, but that critical defects cannot be introduced after Mar-Straining. Since this area appeared to be the most critical, the effect of notches introduced after pre-straining and aging was studied.

A recent report by the ASTM Committee on Fracture Testing of High Strength Sheet Materials (5) has discussed the behavior of an existing crack in a structure in terms of the parameter K, which is defined as a stress field intensity factor. The behavior of the crack, whether it

remains at its original size, grows larger gradually, or propagates rapidly, will depend upon the characteristics of the actual elastic stress field which surrounds it and on the material properties. The actual elastic stress field is the result of the modification of the nominal stress field by the presence of the crack. The quantity representing the combined effect of the crack dimensions and the nominal stress field which influences the behavior of the crack is the stress intensity factor, K.

If loads are applied to any structure containing a crack so that tensile stresses act to open the crack, the value of K increases linearly with the value of the nominal tensile stress component normal to the crack. As the value of K increases, some point will be reached at which the crack will start to increase in length. This point will depend on the material. At first the crack extension is self-limiting. It will not continue unless the nominal stress continues to increase. This is a consequence of plastic deformation which occurs in the vicinity of the advancing crack front, in effect increasing the resistance to further extension of the crack. In this stage,each small increment of stress which causes the crack to extend further also increases the plastic zone size, and hence increases the resistance to crack extension, so that the crack extension per small unit increment of stress is limited. However, the amount of crack extension per unit of stress increment increases as the stress increases, and eventually the process ceases to be self-limiting, so that the crack extension continues without further increases in stress, resulting in unstable rapid crack propagation.

The two major events in this sequence are the start of crack extension

(slow crack growth) and the onset of rapid crack propagation. The toughness of an alloy can be expressed at these two stages or at some value in between. In order to calculate a value for K,two values are required, the length of the crack and the nominal stress acting normal to the crack.. The stress is easily calculated from the specimen configuration and the applied load. The measurement of crack length is more complex. The extension of the crack can be recorded by high speed camera, but this complicates the test procedure. The generally accepted K value used to quantitatively express the toughness of materials and to assist in analysis of failure because of cracks is the Kc quantity which is the K value at onset of rapid crack propagation. The length of crack at the onset of rapid propagation is usually measured by ink staining. India ink is placed in the machined crack prior to stressing. The ink is able to follow the crack growth as it moves slowly but stops at the onset of rapid fracture. The length of ink stain plus the machined notch is the length required for calculation. The load at fracture of the specimen is used to calculate the nominal stress. A second useful value is the K value at the initiation of slow crack growth, $K_{Ic}$. Kc is equal to $K_{Ic}$ when the material and/or specimen configuration is such that slow crack growth does not occur. The absence of slow crack growth is indicative of a completely brittle failure which is void of or has negligible plastic flow associated with the failure and is usually associated with flat cleavage fracture along the full length of the fractured surface. Fractures preceded by slow crack growth have varying amounts of angular shear surfaces associated with rapid crack propagation.

## Procedure

In order to determine accurately the Kc value, certain requirements for specimen dimensions have been established (6). A width to thickness of at least 16 to 1 must be maintained. Length of the specimen and length of the center notch are also expressed in terms of the required specimen width. To determine the notch strength of Mar-Strained material, the specimen design must be such as to allow pre-straining to 1.0% before machining of the center notch so that the effect of pre-strain can be determined. A specimen was designed with the dimensions as shown in Figure 45. The large specimen tabs were required to allow pre-straining to stresses up to 325,000 psi across the gage section without failure of the tab area or fracture of the pins.

The specimens were rough machined to this configuration and then heat treated. Heat treatments used were identical to those which were previously evaluated. After heat treatment, the specimens were ground to final thickness, pre-strained, aged and then notched. Modified S-5 was pre-strained .2, .4, .6, .8 and 1.0 nominal percentages. D6AC was pre-strained .2, .4, .6, and .8%. After notching, the specimens were tested, using ink staining to mark the slow crack growth, and Kc values calculated. The procedure outlined in (6) was used to calculate the Kc values.

## Results

The results obtained for Mar-Strained D6AC and Modified S-5 are found in Tables 5 and 6. The strength values reported are (1) notch strength-breaking load divided by the area of the specimen minus the original crack

CENTER NOTCH SPECIMEN

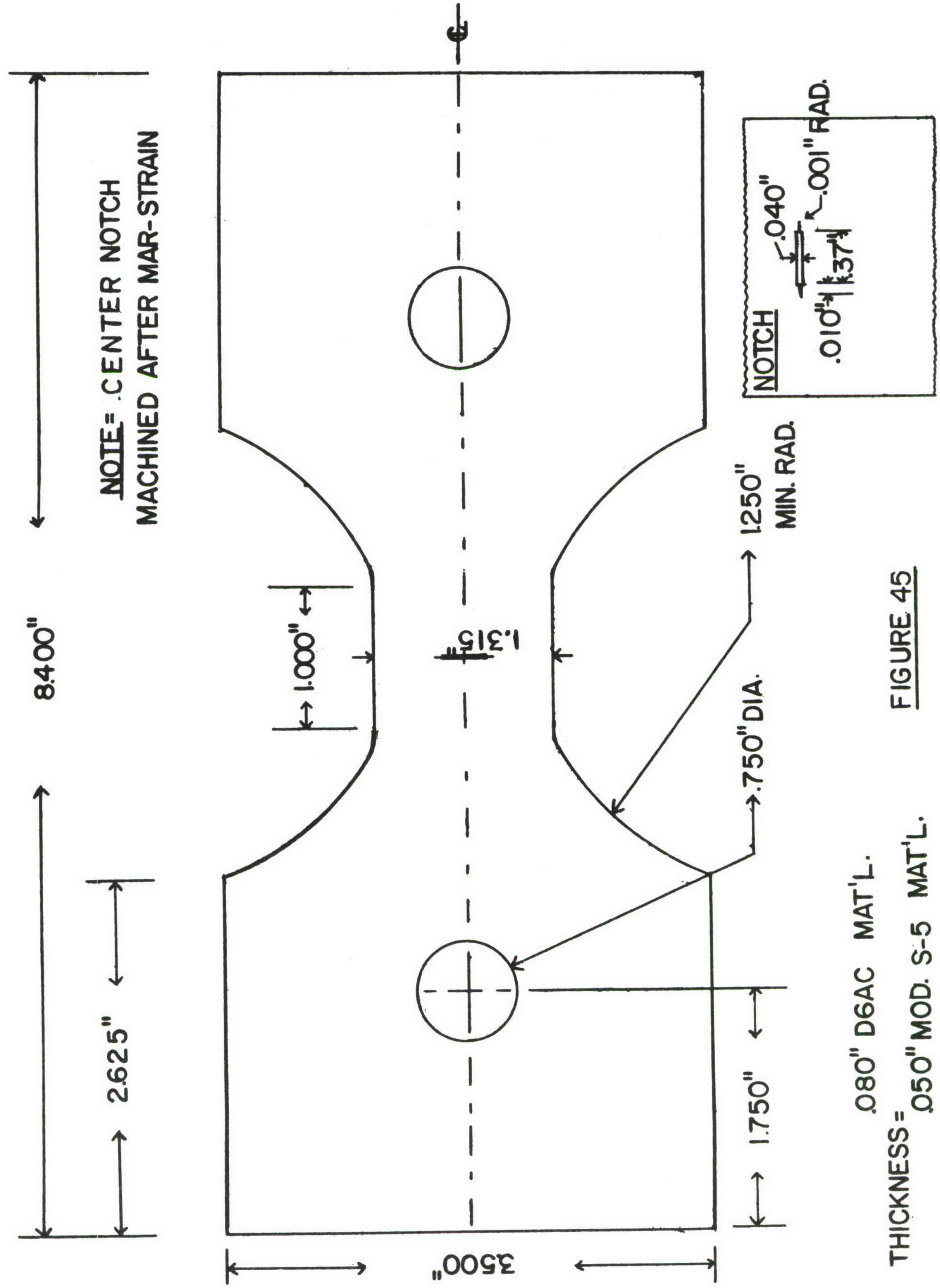



FIGURE 45

## TABLE 5

## CENTER NOTCH DATA FOR MAR-STRAINED LADISH D6AC

| Specimen Number | % Pre-Strain | Notch Str. KSI | Gross Str. KSI | 2a-inches | a-inches | $\frac{\pi a}{W}$ | Y.S. KSI | $\left(\frac{\text{Gross}}{\text{Y.S.}}\right)^2$ | Q1 | Kc | Net Fracture Str. | Specimen Width - in. | Specimen Thickness -in. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 0 | 138 | 95 | .65 | .325 | .778 | 240 | .157 | 1.2 | 119 | 189 | 1.3 | .080 |
| 13 | 0 | 144 | 99.3 | .60 | .30 | .715 | 240 | .173 | 1.03 | 115 | 180 | 1.3 | .080 |
| 14 | 0 | 124 | 85.7 | .64 | .32 | .764 | 240 | .127 | 1.08 | 102 | 167 | 1.3 | .080 |
| Aver. | | 135 | 93.3 | .63 | | | | | | 112 | 175 | | |
| 2 | .20 | 127 | 87 | .58 | .29 | .695 | 262 | .110 | .95 | 97.5 | 156 | 1.3 | .080 |
| 3 | .24 | 117.5 | 80.7 | .50 | .25 | .598 | 262 | .092 | .75 | 80.0 | 131 | 1.3 | .080 |
| Aver. | | 122 | 83.8 | .54 | | | | | | 88.7 | 143 | | |
| 4 | .40 | 112.3 | 77.7 | .58 | .29 | .698 | 277 | .079 | .9 | 84.5 | 140 | 1.3 | .080 |
| 5 | .40 | 121.7 | 84.0 | .68 | .34 | .808 | 277 | .092 | 1.15 | 103 | 174 | 1.3 | .080 |
| 6 | .40 | 121.3 | 83.7 | .60 | .30 | .725 | 277 | .0915 | .95 | 93 | 156 | 1.3 | .080 |
| Aver. | | 118.4 | 81.8 | .62 | | | | | | 93.5 | 157 | 1.3 | .080 |
| 7 | .62 | 117.3 | 80.5 | .68 | .34 | .817 | 285 | .0857 | 1.15 | 98.8 | 168 | 1.3 | .080 |
| 8 | .60 | 134.5 | 92.8 | .70 | .35 | .838 | 285 | .166 | 1.25 | 117 | 202 | 1.3 | .080 |
| 9 | .62 | 134.2 | 92.5 | .64 | .32 | .770 | 285 | .106 | 1.1 | 111 | 181 | 1.3 | .080 |
| Aver. | | 128.7 | 88.6 | .67 | | | | | | 108.9 | 187 | | |
| 10 | .80 | 121.2 | 83.7 | .62 | .31 | .745 | 288 | .0845 | 1.05 | 86 | 159 | 1.3 | .080 |
| 11 | .80 | 110 | 76.8 | .62 | .31 | .745 | 288 | .069 | 1.00 | 87.5 | 147 | 1.3 | .080 |
| 12 | .81 | 116 | 80.3 | .62 | .31 | .745 | 288 | .078 | 1.0 | 91.5 | 153 | 1.3 | .080 |
| Aver. | | 116 | 80.3 | .62 | | | | | | 88.3 | 153 | | |

# TABLE 6

## CENTER NOTCH DATA FOR MAR-STRAINED MODIFIED S-5

| Specimen Number | % Pre-Strain | Notch Str. KSI | Gross (Nominal) Str. KSI | 2a-inches | a | $\frac{\pi a}{W}$ | Y.S. KSI | $\left(\frac{Gross}{Y.S.}\right)^2$ | Ql | Kc | Net Fracture Str. KSI | Specimen Width -in. | Specimen Thickness -in. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 11 | 0 | 171.2 | 121.5 | .66 | .33 | .786 | 280 | .188 | 1.25 | 156 | 240 | 1.3 | .050 |
| 18 | 0 | 153.0 | 116.8 | .55 | .275 | .657 | 280 | .173 | .9 | 127 | 183 | 1.3 | .050 |
| 17 | 0 | 178.5 | 124.0 | .66 | .33 | .783 | 280 | .196 | 1.25 | 160 | 248 | 1.3 | .050 |
| Aver. | | 167.6 | 120.7 | .62 | | | | | | 144 | 223 | | |
| 1 | .21 | 113 | 78.3 | .48 | .24 | .574 | 298 | .069 | .7 | 75.2 | 121.5 | 1.3 | .050 |
| 2 | .23 | 101.2 | 70.3 | .54 | .27 | .645 | 298 | .051 | .8 | 72.2 | 119.0 | 1.3 | .050 |
| Aver. | | 107 | 74.3 | .51 | | | | | | 73.7 | 120.3 | | |
| 5 | .41 | 134 | 93.8 | .55 | .275 | .657 | 308 | .092 | 1.08 | 97.6 | 162 | 1.3 | .050 |
| 4 | .42 | 145.8 | 101.5 | .48 | .24 | .574 | 308 | .108 | 0.7 | 97.6 | 157.5 | 1.3 | .050 |
| 6 | .46 | 107.5 | 74.6 | .64 | .32 | .764 | 308 | .0586 | 1.0 | 85.5 | 147.0 | 1.3 | .050 |
| Aver. | | 129.1 | 89.9 | .55 | | | | | | 93.6 | 155.3 | | |
| 7 | .60 | 126.5 | 88.3 | .55 | .275 | .660 | 318 | .077 | .82 | 91.8 | 152.0 | 1.3 | .050 |
| 8 | .60 | 125.8 | 87.3 | .54 | .27 | .645 | 318 | .076 | .8 | 88.5 | 147.0 | 1.3 | .050 |
| 9 | .62 | 109.6 | 75.8 | .58 | .29 | .694 | 318 | .057 | .9 | 82.3 | 143.5 | 1.3 | .050 |
| Aver. | | 120.6 | 83.8 | .56 | | | | | | 87.5 | 147.3 | | |
| 10 | .82 | 127.2 | 88.1 | .52 | .26 | .623 | 323 | .075 | .75 | 87.2 | 146.5 | 1.3 | .050 |
| 16 | .84 | 106.6 | 74.7 | .48 | .24 | .576 | 323 | .053 | .67 | 70.0 | 115.5 | 1.3 | .050 |
| 12 | .81 | 132 | 91.5 | .55 | .275 | .658 | 323 | .08 | .83 | 96.5 | 157 | 1.3 | .050 |
| Aver. | | 121.9 | | .52 | | | | | | 84.2 | 139.7 | | |
| 15 | 1.01 | 129 | 98.9 | .65 | .325 | .783 | 326 | .092 | 1.05 | 116 | 218 | 1.3 | .050 |
| 13 | 1.02 | 123 | 85.8 | .57 | .285 | .680 | 326 | .069 | .88 | 92.3 | 150.5 | 1.3 | .050 |
| 14 | 1.02 | 113 | 79.2 | .55 | .275 | .658 | 326 | .059 | .80 | 81.3 | 136.5 | 1.3 | .050 |
| Aver. | | 121.6 | 87.9 | .59 | | | | | | 96.5 | 168 | | |

area, (2) the gross notch or nominal strength - the breaking load divided by the original area, (3) the net fracture strength -the breaking load divided by the original specimen area minus the sum of the area of the machined notch plus the area of slow crack growth. The 2a value tabulated is the length of the slow crack growth including the machined notch. The percent pre-strain given each specimen is also shown.

As is true of most notch testing, considerable spread in the data was experienced, of the order of 10%. This amount of spread existed for nearly all amounts of pre-strain. In Figures 46A and 46B the Kc values for the two alloys are shown versus percent pre-strain. The notch strengths are shown in Figure 47A and 47B. It can be readily noted by examination of these figures that the addition of pre-straining and aging was detrimental to the notch toughness of these two low-alloy steels, but that this detrimental effect was not further aggravated by increasing the amount of strain. The loss in toughness was experienced after only .2% pre-strain. The values of Kc and notch strength of Mar-Strained D6AC and Modified S-5 were very nearly equal even though the specimens varied in thickness (.050 for S-5 and .080 for D6AC) and the alloys differed in yield strength and hardness. The decrease in notch toughness was more apparent with the Modified S-5 alloy than the D6AC primarily because of the greater toughness obtained with the S-5 alloy in the as heat treated (0% pre-strain) condition.

In first viewing the results, it would appear that Mar-Straining has decreased the Kc value to the $K_{I_c}$ value (a condition of extreme brittleness), but the slow crack growth (2a values in Tables 5 and 6) has not been eliminated and is very nearly equal to the amount of growth experienced in the

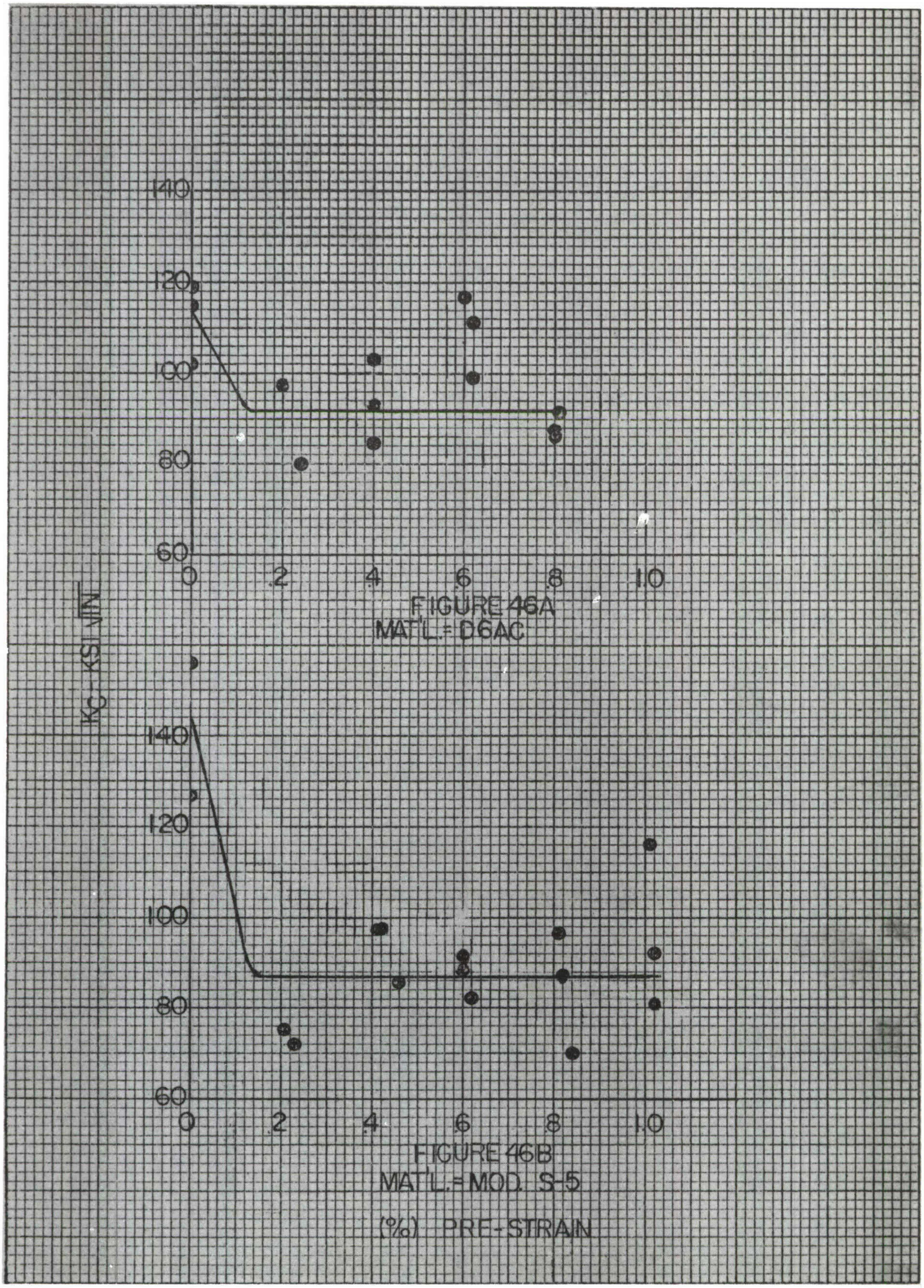



FIGURE 46

NOTCH TOUGHNESS ($K_C$) FOR MAR-STRAINED ALLOYS

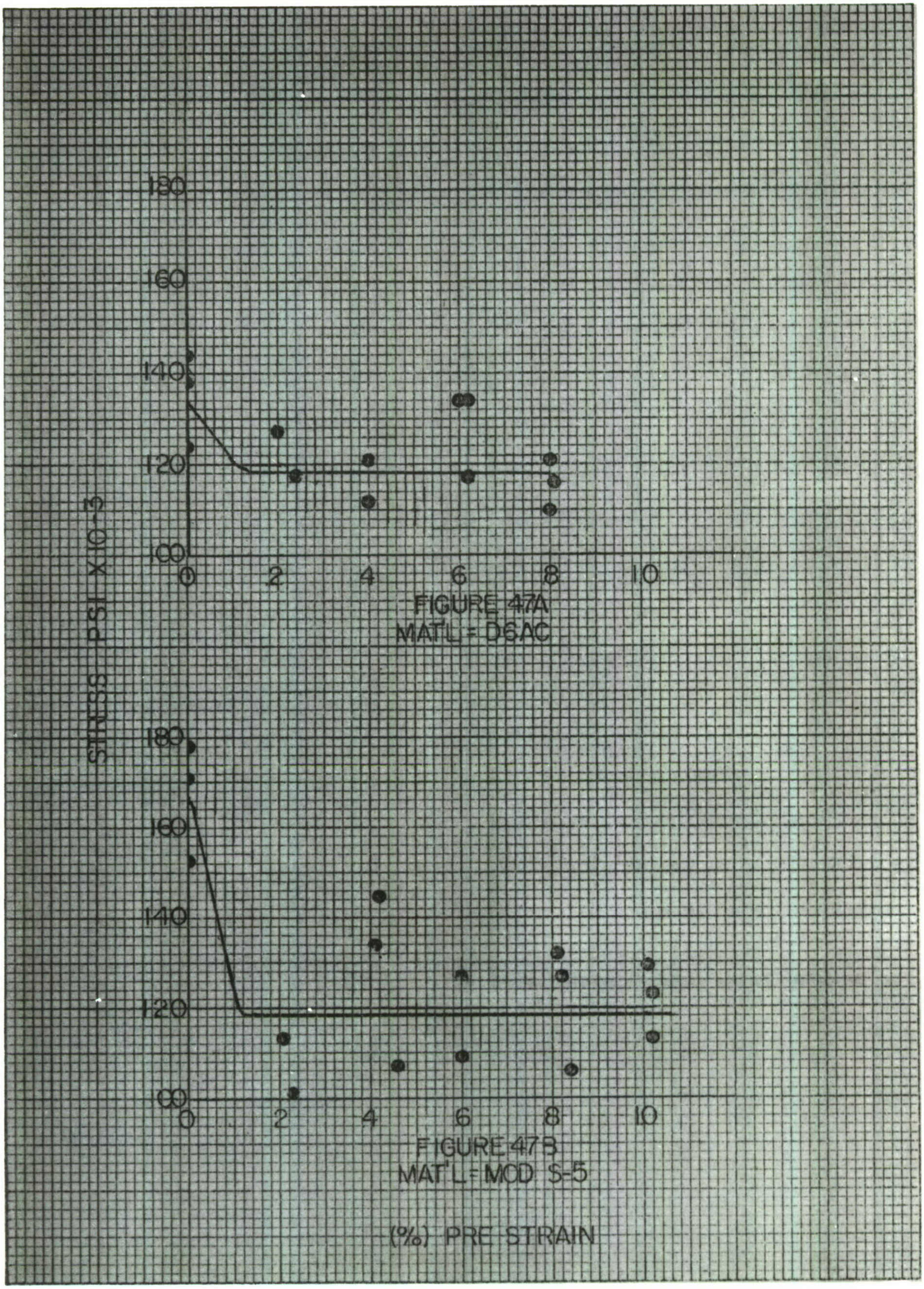



FIGURE 47
NOTCH STRENGTH OF MAR-STRAINED ALLOYS

as heat treated alloys. A further explanation was required to explain the decrease in toughness. Crack propagation theory states that the average uniform stress acting normal to the crack (gross stress) is related to the K value by the expression.

$$\sigma = \frac{K}{\sqrt{\pi a}}$$

where $\sigma$ = gross stress

$a$ = 1/2 of crack length

if the effect of plastic strain is neglected; also $\sigma$ and K are numerically equal when $a = \left(\frac{1}{\pi}\right)$. The data obtained indicate that the relationship defined by the above equation existed. This implies that the plastic strain which occurred did not act to increase the Kc value to any great degree (Kc values are approximately 10% higher than that calculated from the above equation) except in the as heat treated material. Previous tensile data has shown that as the amount of pre-straining is increased the Mar-Strain yield strength approaches and becomes equal to the ultimate strength bringing about a decrease in the strain hardening capacity of the alloy. The decrease in notch toughness experienced is believed to have been caused by this decrease in strain hardening. It must also be realized that this condition of lower fracture toughness pertains to large cracks introduced into a Mar-Strained specimen after straining and aging. As has been previously discussed, the Mar-Strain process is expected to be beneficial in increasing the toughness associated with defects originally present in the as heat treated structure by the addition of compressive stresses and/or notch blunting during the pre-straining process. Nevertheless, the values of $K_c$ obtained are higher than some hot work die steels (H-11) which have been successfully used in pressure vessels.

## E.) Fatigue Testing

The fatigue portion of the engineering property evaluation was planned to provide the following comparisons and information.

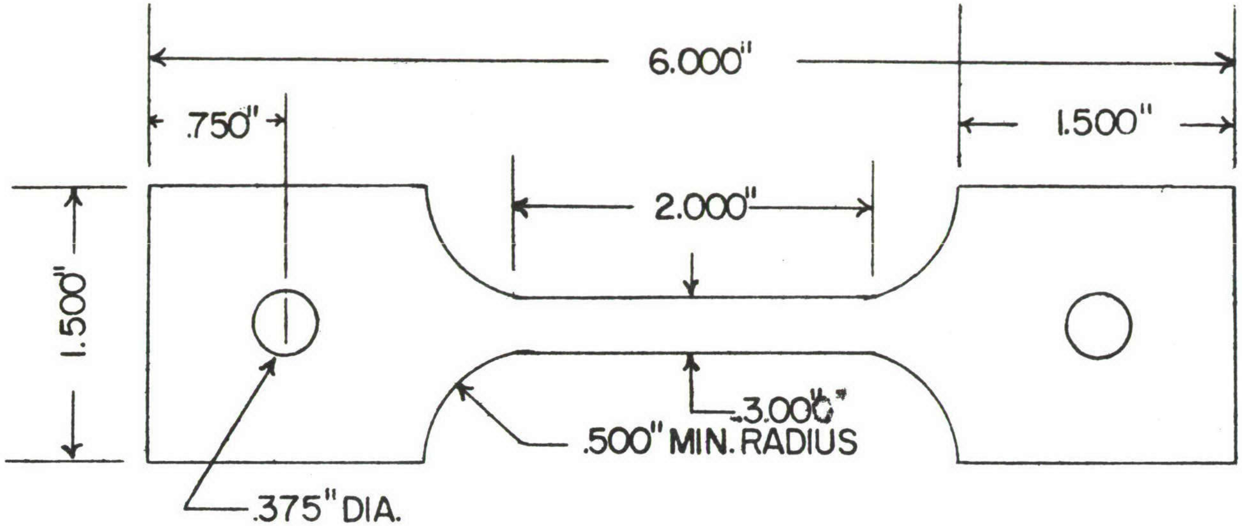
1) The effect of Mar-Straining on the fatigue properties of D6AC and Modified S-5 at low cycle-high stress and high cycle-low stress conditions.

2) To provide sufficient data to statistically analyze the upper knee and the run-out portion of the S-N curves.

3) To provide a comparison of the run-out stress for specimens fatigued under tension-tension versus alternating stress (bending) conditions.

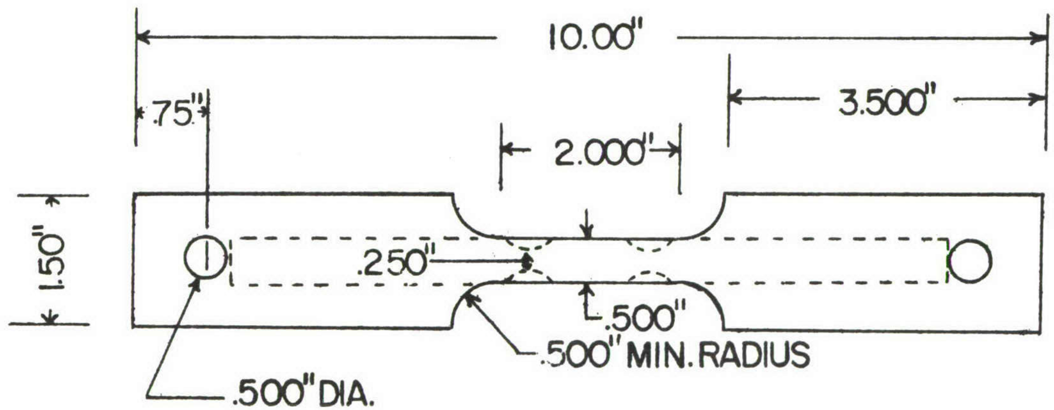
Procedure -

Strips, 1.5 inches X 6.5 inches or 1.5 X 10 inches, were cut from sheet stock transverse to the final direction of rolling. The strips were then spheroidize annealed and machined to the configurations shown in Figure 48. The specimens were then heat treated as was described under the tensile property determinations. One half of the specimens of each alloy was designated for evaluation in the as heat treated condition and ground to the final thickness and finish (as described in Figure 48). The remaining half was ground to within .010 inches of the final thickness, pre-strained and aged. The Modified S-5 was pre-strained .28% and the D6AC .38%. These values of pre-strain were determined from the tensile studies to give the desired yield strengths (275,000 psi - D6AC and 300,000 psi - Modified S-5). After aging, the Mar-Strained specimens were ground to final thickness and surface finish. All specimens were stored in mineral oil until tested to eliminate degradation of properties




TENSION-TENSION FATIGUE SPECIMEN

THICKNESS .050" MOD. S-5
.080" D6AC




NOTE = 16 RMS FINISH GAGE SECTION

ALTERNATING STRESS FATIGUE SPECIMEN
SCALE 1/2 = 1

FIGURE 48

by surface corrosion.

Up to 3500 cycles, tension-tension data was obtained on a 10,000 pound Instron tensile machine. For the higher cycle portion of the curve (up to $10^7$) data was obtained on a 9000 pound capacity Ivy (Model BJL-1) combined stress fatigue testing machine (Figure 49). A dynamic to static stress ratio of .90 to .96 was maintained on both machines. Several strain gaged specimens were tested to determine if this ratio was maintained during the test and to make certain compressive stresses were not incurred.

The alternating stress data was obtained on an electromagnetic sheet fatigue machine[7]. Specimens were designed to provide two tests from each specimen.

Results -

Modified S-5 - The tension-tension properties of Modified S-5 are tabulated in Table 7 and are plotted in Figures 50, 51, 52 and 53. Figure 50 was prepared to show the overall shape of the S/N curve for the alloy and to allow rapid comparison of the two conditions. This figure clearly shows the increase in stress (at any number of cycles) afforded by Mar-Straining the alloy. In the low cycle region, the Mar-Strain curve remained at a nearly constant stress which was approximately equal to the ultimate tensile strength. The as heat treated curve had a gradual slope downward in stress as the number of cycles increased. The knees of both curves were observed to begin at approximately the yield strength of the condition-300,000 psi for Mar-Strained and 280,000 for the as heat treated. In the run-out portion of the curve the Mar-Strained condition again demonstrated its superiority.

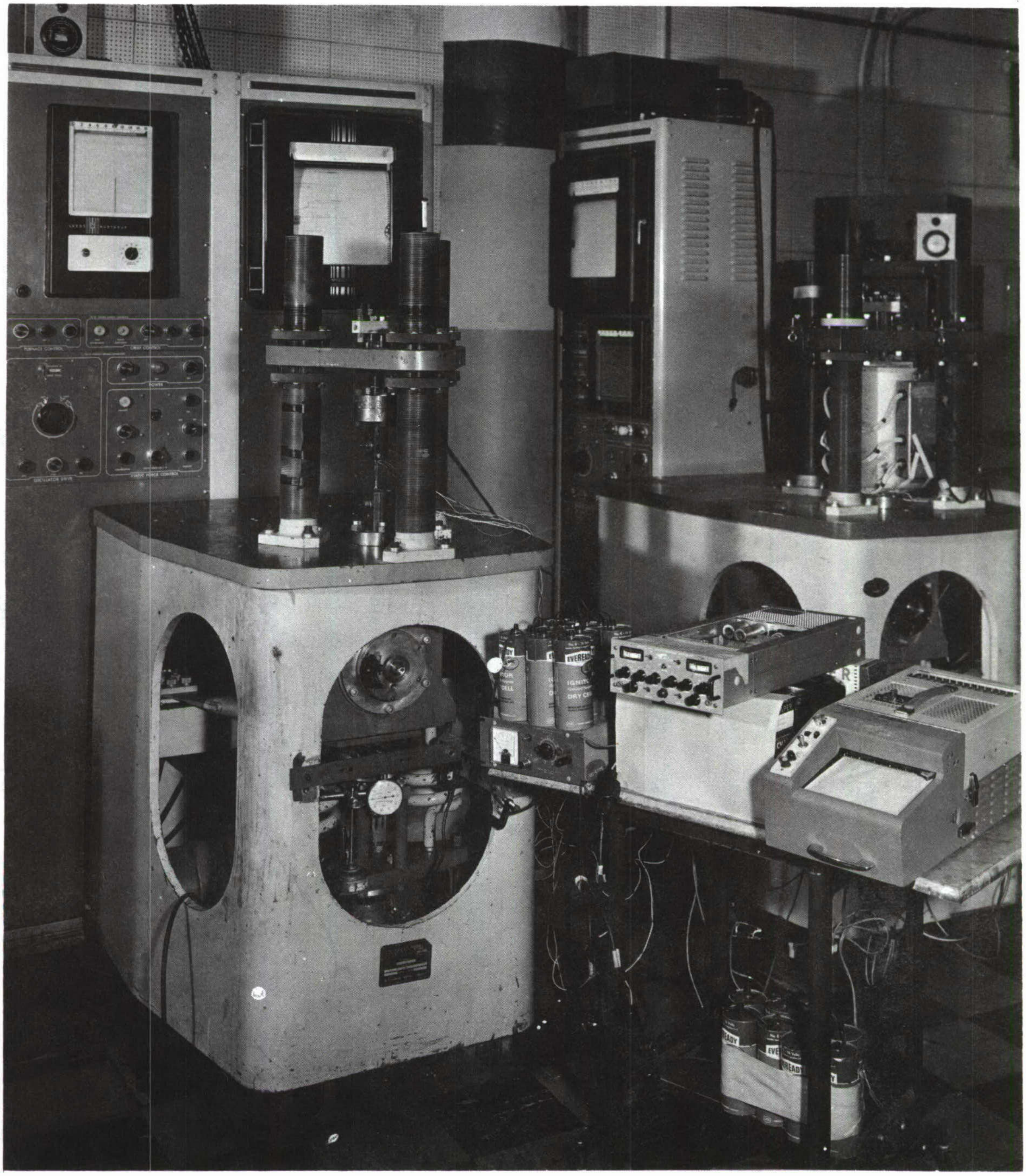


Figure 49. 9000 Pound Capacity Ivy (Model BJL-1) Combined Stress Fatigue Testing Machine with Strain Gage Monitoring Equipment.

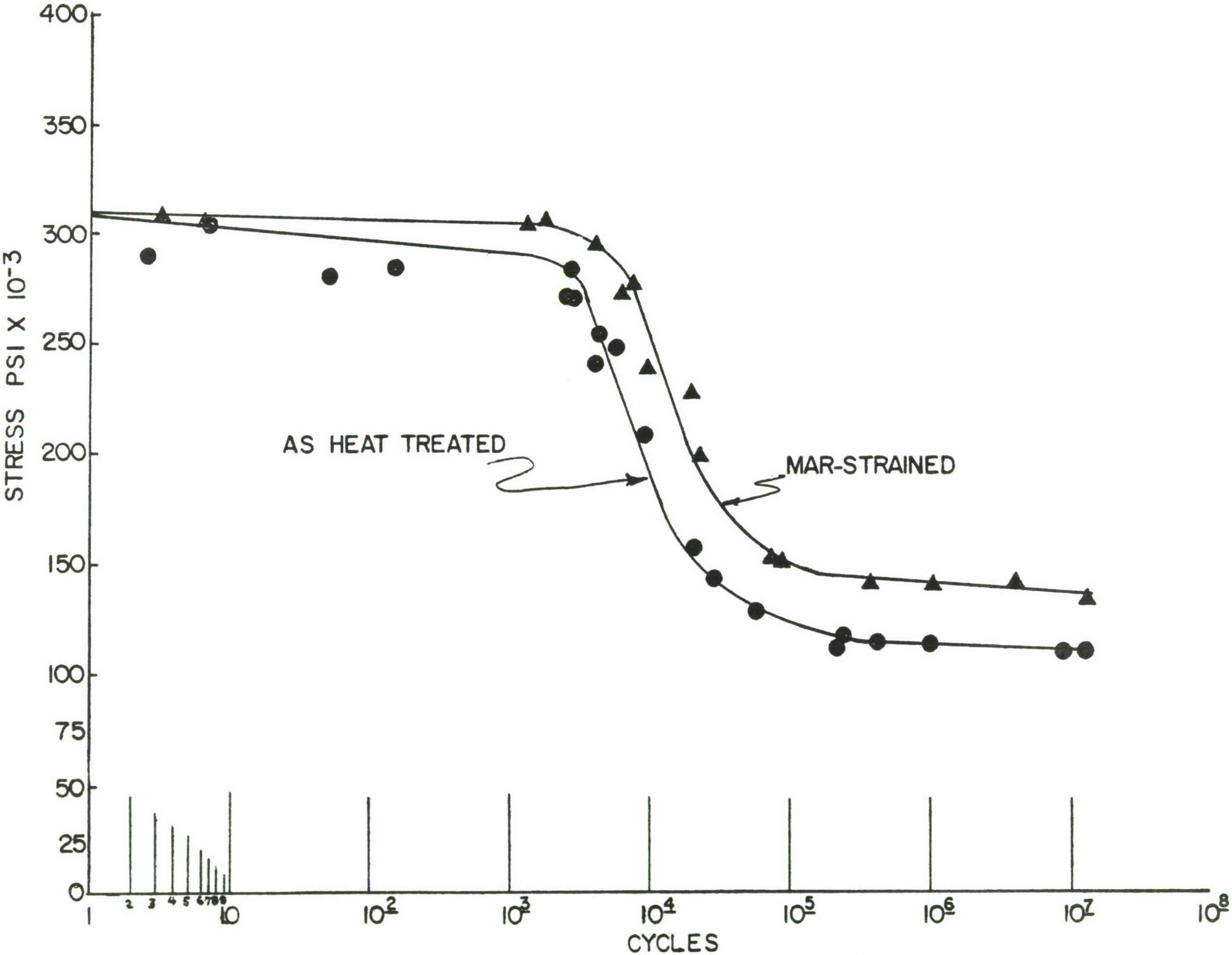



FIGURE 50. AXIAL FATIGUE PROPERTIES MODIFIED S-5

TABLE 7

TENSION-TENSION FATIGUE PROPERTIES OF MODIFIED S-5

Material = Modified S-5 - As Heat Treated Condition

| Machine Type | Spec. No. | No. of Cycles | Stress |
|---|---|---|---|
| Instron | 1A | 1 | 294,000 Psi |
| Instron | 2A | 3 | 294,000 Psi |
| Instron | 3A | 8 | 309,000 Psi |
| Instron | 4A | 750 | 282,000 Psi |
| Instron | 5A | 1750 | 284,000 Psi |
| Ivy | 10 | 3000 | 272,500 Psi |
| Ivy | 32 | 3200 | 285,000 Psi |
| Ivy | 5 | 3300 | 270,000 Psi |
| Ivy | 6 | 4500 | 240,000 Psi |
| Ivy | 25 | 4500 | 262,400 Psi |
| Ivy | 31 | 6500 | 247,500 Psi |
| Ivy | 3 | 10,000 | 210,300 Psi |
| Ivy | 4 | 23,000 | 157,000 Psi |
| Ivy | 5 | 33,000 | 145,000 Psi |
| Ivy | 8 | 64,000 | 130,000 Psi |
| Ivy | 13 | 230,000 | 113,000 Psi |
| Ivy | 2 | 264,000 | 120,000 Psi |
| Ivy | 11 | 336,000 | 115,000 Psi |
| Ivy | 9 | 1,010,000 | 117,500 Psi |
| Ivy | 7 | 2,242,000 | 115,000 Psi |
| Ivy | 14 | 10,018,000 + | 110,000 Psi |
| Ivy | 12 | 13,521,000 + | 110,000 Psi |

+No failure.

TABLE 7 (Continued)

**Material = Modified S-5 - Mar-Strained Condition**

| Machine Type | Spec. No. | No. of Cycles | Stress |
|---|---|---|---|
| Instron | 16 | 9 | 305,000 Psi |
| Instron | 11 | 4 | 309,000 Psi |
| Instron | 4 | 1200 | 310,000 Psi |
| Instron | 19 | 1560 | 312,000 Psi |
| Ivy | 22 | 3250 | 301,500 Psi |
| Ivy | 3 | 6000 | 270,000 Psi |
| Ivy | 6 | 7000 | 280,000 Psi |
| Ivy | 18 | 10,000 | 240,000 Psi |
| Ivy | 1 | 22,500 | 220,000 Psi |
| Ivy | 27 | 25,000 | 200,000 Psi |
| Ivy | 9 | 68,500 | 148,000 Psi |
| Ivy | 28 | 69,000 | 148,000 Psi |
| Ivy | 2 | 74,000 | 155,000 Psi |
| Ivy | 7 | 82,000 | 145,000 Psi |
| Ivy | 30 | 82,500 | 142,000 Psi |
| Ivy | 29 | 424,000 | 146,000 Psi |
| Ivy | 17 | 1,103,000 | 146,000 Psi |
| Ivy | 8 | 4,567,000 | 145,000 Psi |
| Ivy | 15 | 13,675,000 + | 135,000 Psi |

+No failure

Figures 51, 52 and 53 were prepared to display the data in greater detail and to present the results obtained in a more useful engineering manner.

Three areas of these curves were investigated statistically. They were from 2000 to 7000 cycles, 20,000 to 70,000 cycles and in the run-out area. In the run-out area, a mean fatigue limit was obtained by a "staircase" analysis. A standard deviation was calculated and an estimate made of the 95% confidence limits. Standard deviations were calculated at the other two areas for the mean stress observation and an average number of cycles. A sample calculation for each of these analyses is found in the Appendix. The results obtained were as follows:

| Area | Heat Treated | Mar-Strained |
|---|---|---|
| 2000 to 7000 | | |
| Mean observation | 263,000 psi | 284,000 psi |
| Average cycles | 4,200 | 5,400 |
| Std. deviation | 15,000 psi | 13,000 psi |
| 20,000 to 70,000 | | |
| Mean observation | 144,000 psi | 179,000 psi |
| Average cycles | 40,000 | 46,000 |
| Std. deviation | 11,000 psi | 32,000 psi |
| Run-Out | | |
| Mean stress | 114,380 psi | 142,900 psi |
| Std. deviation | 7,000 psi | 14,500 psi |
| 95% confidence limits | 120,900 and 107,900 psi | 157,700 and 128,000 psi |

These analyses showed that in the areas chosen, the Mar-Strained condition was prone to more deviation than the heat treated condition, but it was capable of higher stresses. The Mar-Strain mean stress at run-out was 25 percent higher than the heat treated condition.

D6AC - The tension-tension fatigue data for Ladish D6AC is tabulated in Table 8 and is plotted in Figures 54, 55, 56, and 57. The overall S/N curve (Figure 54) has the same general shape as the Modified S-5 alloy S/N curve.

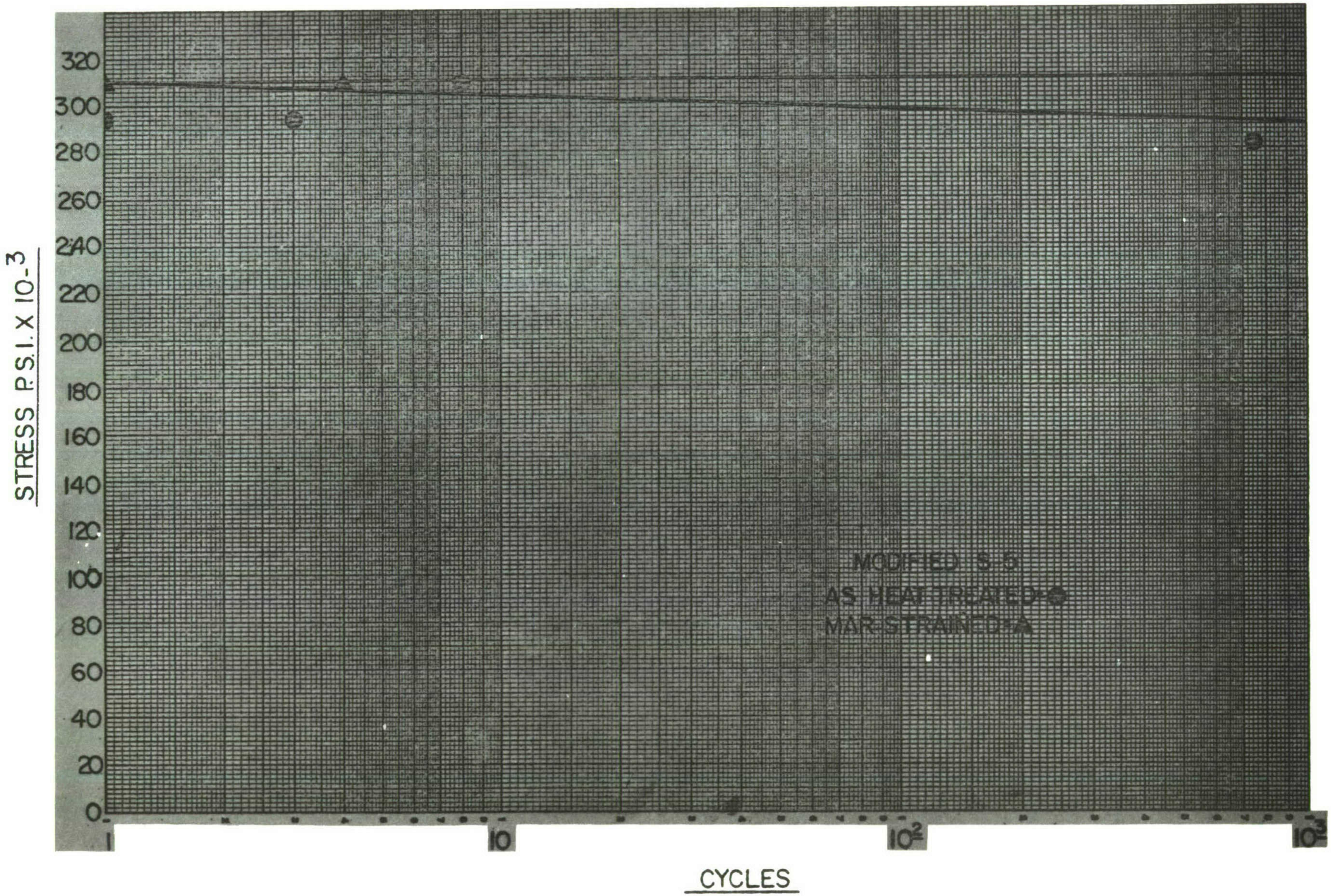



FIGURE 51. AXIAL FATIGUE PROPERTIES MODIFIED S-5

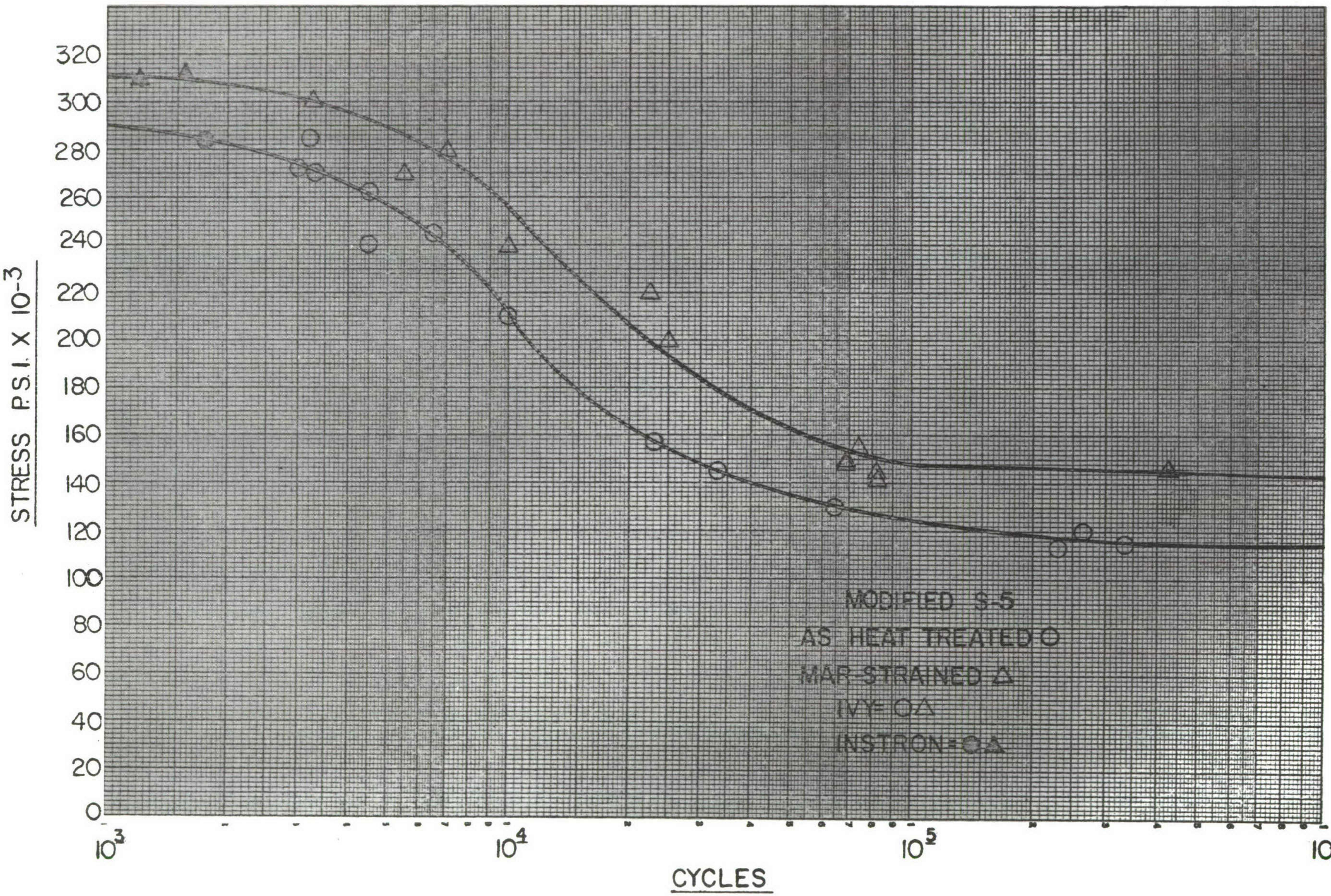



FIGURE 52. AXIAL FATIGUE PROPERTIES MODIFIED S-5

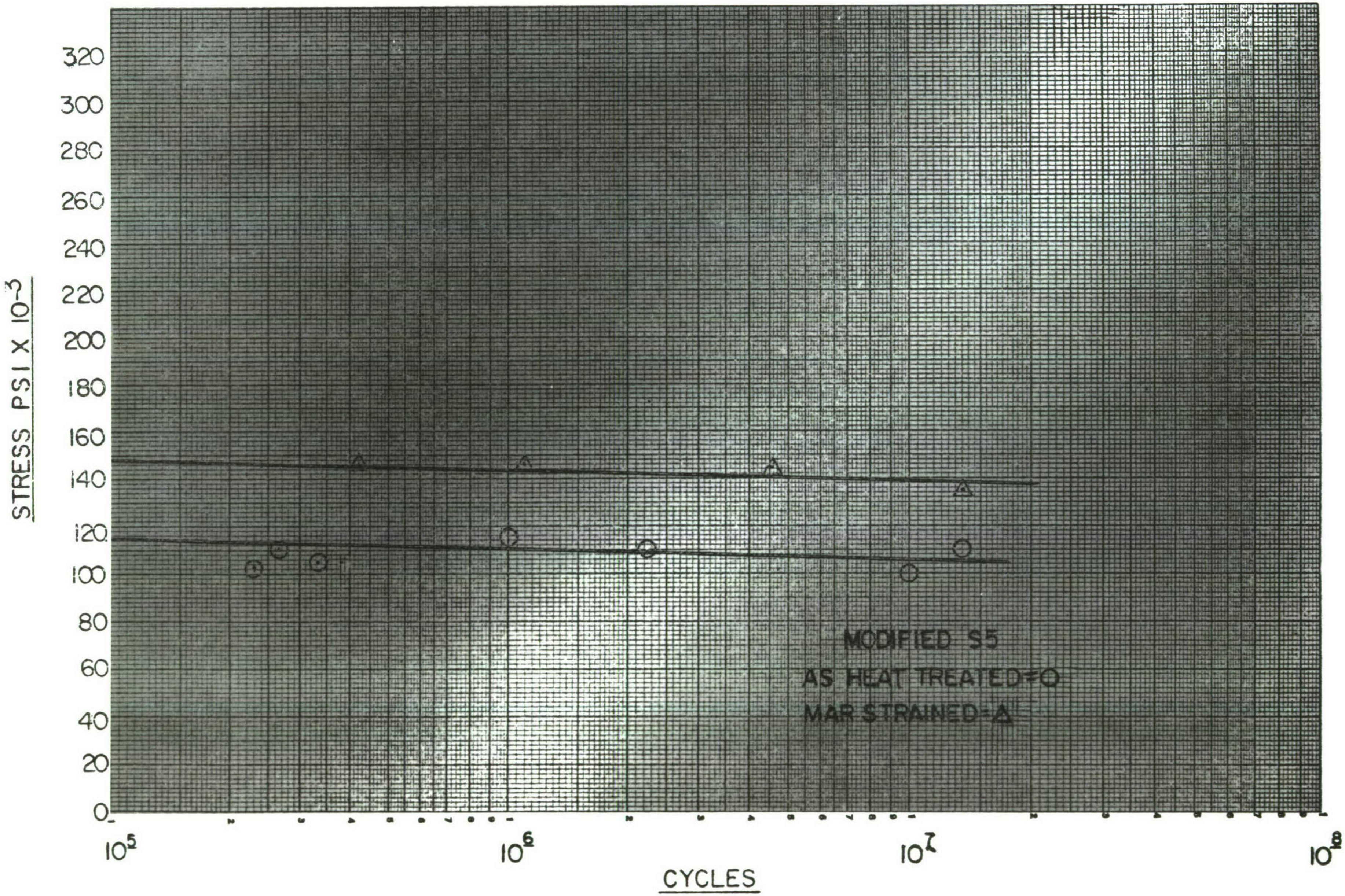



FIGURE 53. AXIAL FATIGUE PROPERTIES MODIFIED S-5

TABLE 8

TENSION-TENSION FATIGUE PROPERTIES OF LADISH D6AC

As Heat Treated

| Machine Type | Spec. No. | Cycles | Stress-Psi |
|---|---|---|---|
| Instron | 1 | 6 | 279,000 |
| Instron | 2 | 6 | 273,000 |
| Instron | 5 | 6 | 271,000 |
| Instron | 3 | 10 | 269,000 |
| Instron | 4 | 3,400 | 264,000 |
| Ivy | 6 | 1,700 | 246,700 |
| Ivy | 7 | 2,400 | 260,000 |
| Ivy | 8 | 2,400 | 255,900 |
| Ivy | 9 | 6,690 | 200,000 |
| Ivy | 10 | 8,600 | 240,000 |
| Ivy | 11 | 11,000 | 216,900 |
| Ivy | 12 | 24,840 | 168,000 |
| Ivy | 13 | 50,000 | 112,000 |
| Ivy | 14 | 88,000 | 140,000 |
| Ivy | 15 | 130,000 | 130,000 |
| Ivy | 16 | 144,000 | 120,000 |
| Ivy | 17 | 160,000 | 108,000 |
| Ivy | 18 | 16,267,000 + | 100,000 |
| Ivy | 19 | 20,398,000 + | 115,000 |
| Ivy | 20 | 24,484,000 + | 104,000 |

Mar-Strained

| Machine Type | Spec. No. | Cycles | Stress-Psi |
|---|---|---|---|
| Instron | 30 | 6 | 270,000 |
| Instron | 26 | 6 | 271,000 |
| Instron | 22 | 7 | 271,000 |
| Instron | 20 | 6 | 273,500 |
| Instron | 23 | 97 | 276,000 |
| Instron | 19 | 2,107 | 274,000 |
| Ivy | 4 | 5,000 | 257,000 |
| Ivy | 7 | 8,000 | 250,000 |
| Ivy | 11 | 13,000 | 230,000 |
| Ivy | 25 | 32,400 | 140,000 |
| Ivy | 9 | 43,200 | 160,000 |
| Ivy | 5 | 118,500 | 150,000 |
| Ivy | 28 | 127,000 | 150,000 |
| Ivy | 1 | 149,000 | 142,000 |
| Ivy | 29 | 169,000 | 145,000 |
| Ivy | 2 | 298,000 | 123,000 |
| Ivy | 27 | 348,000 | 130,000 |
| Ivy | 24 | 788,400 | 135,000 |
| Ivy | 7 | 3,520,000 | 120,000 |
| Ivy | 11 | 14,147,000 + | 140,000 |
| Ivy | 3 | 22,282,000 + | 120,000 |

+No failure

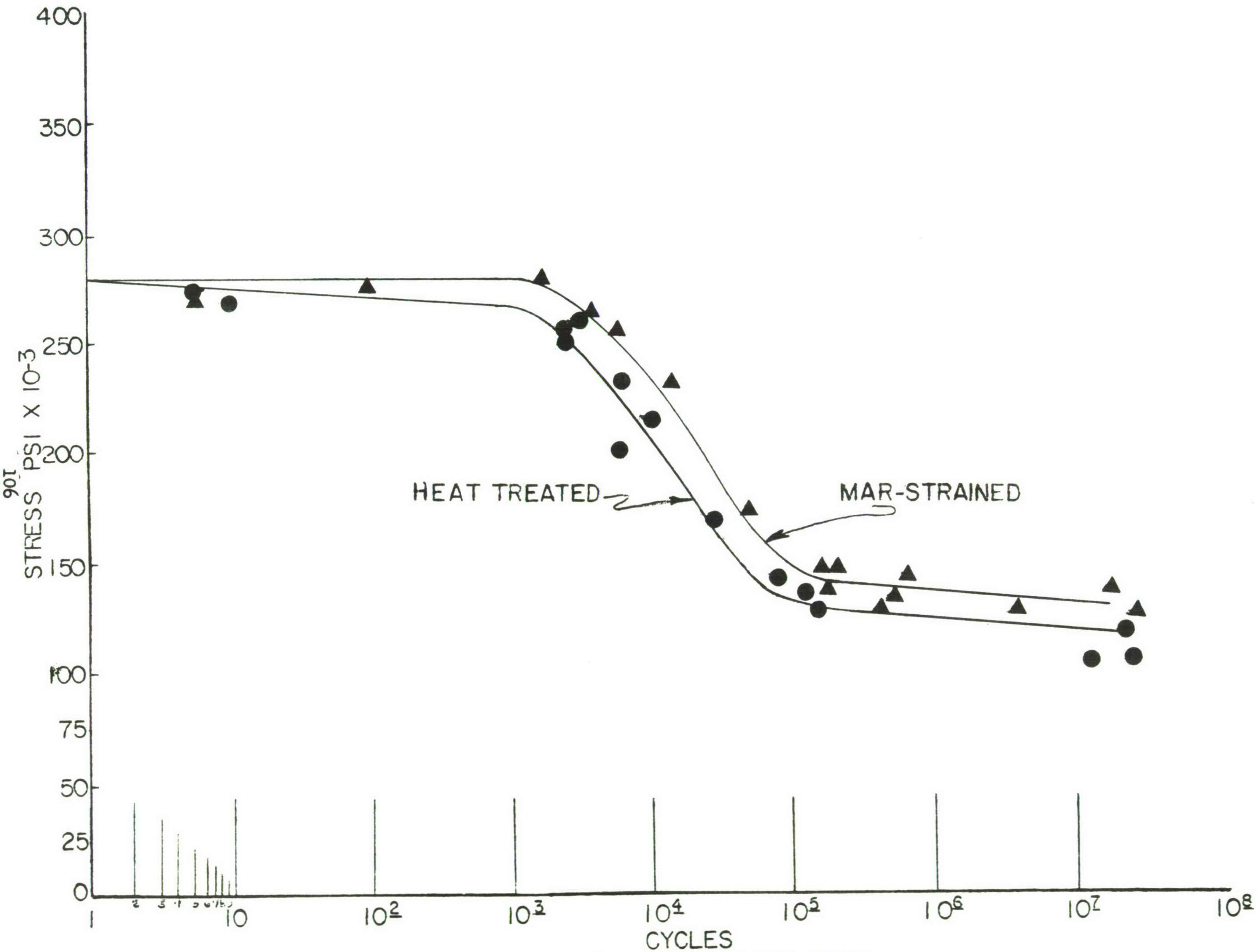



FIGURE 54. AXIAL FATIGUE PROPERTIES D6AC

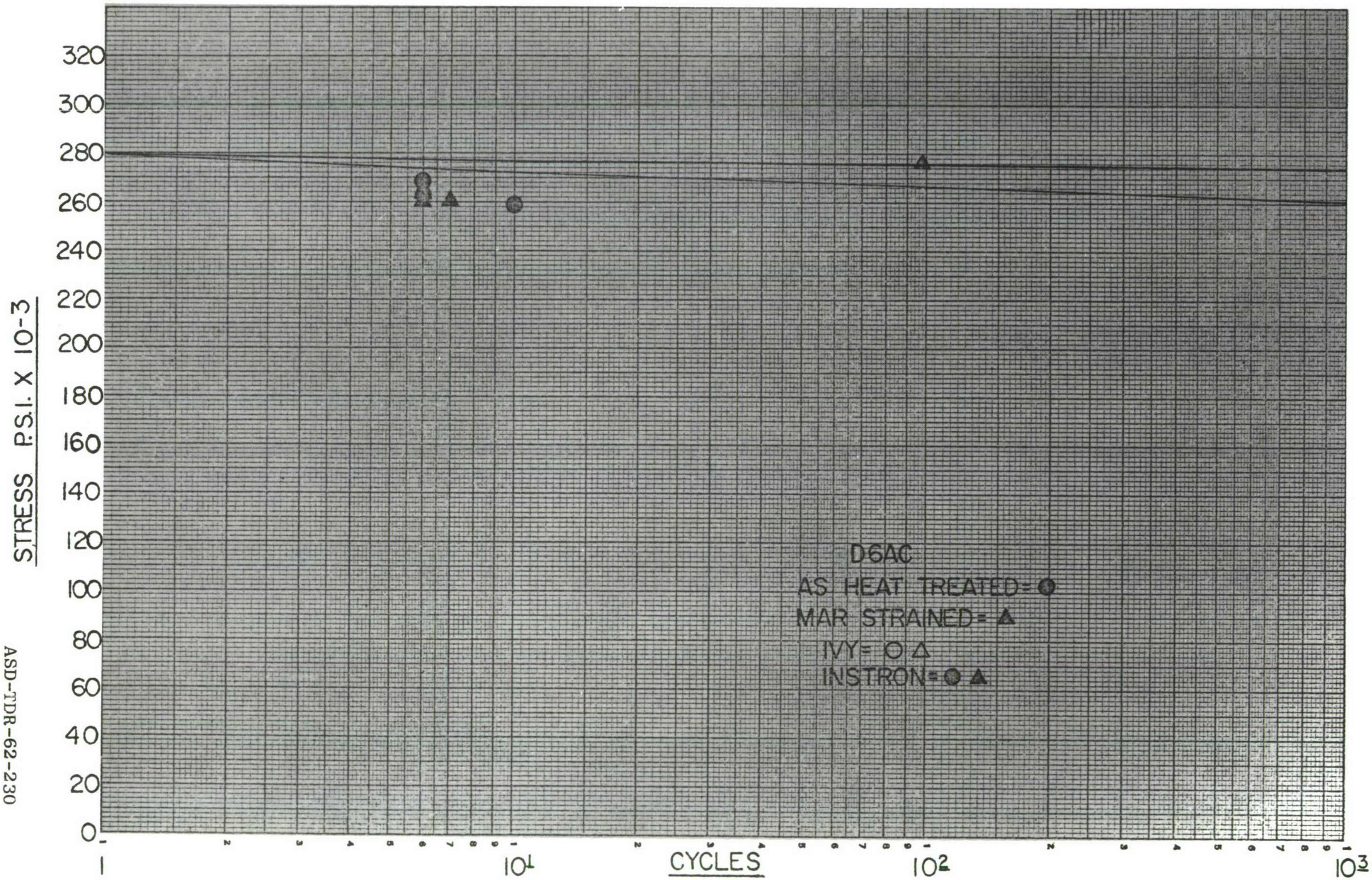



FIGURE 55. AXIAL FATIGUE PROPERTIES D6AC

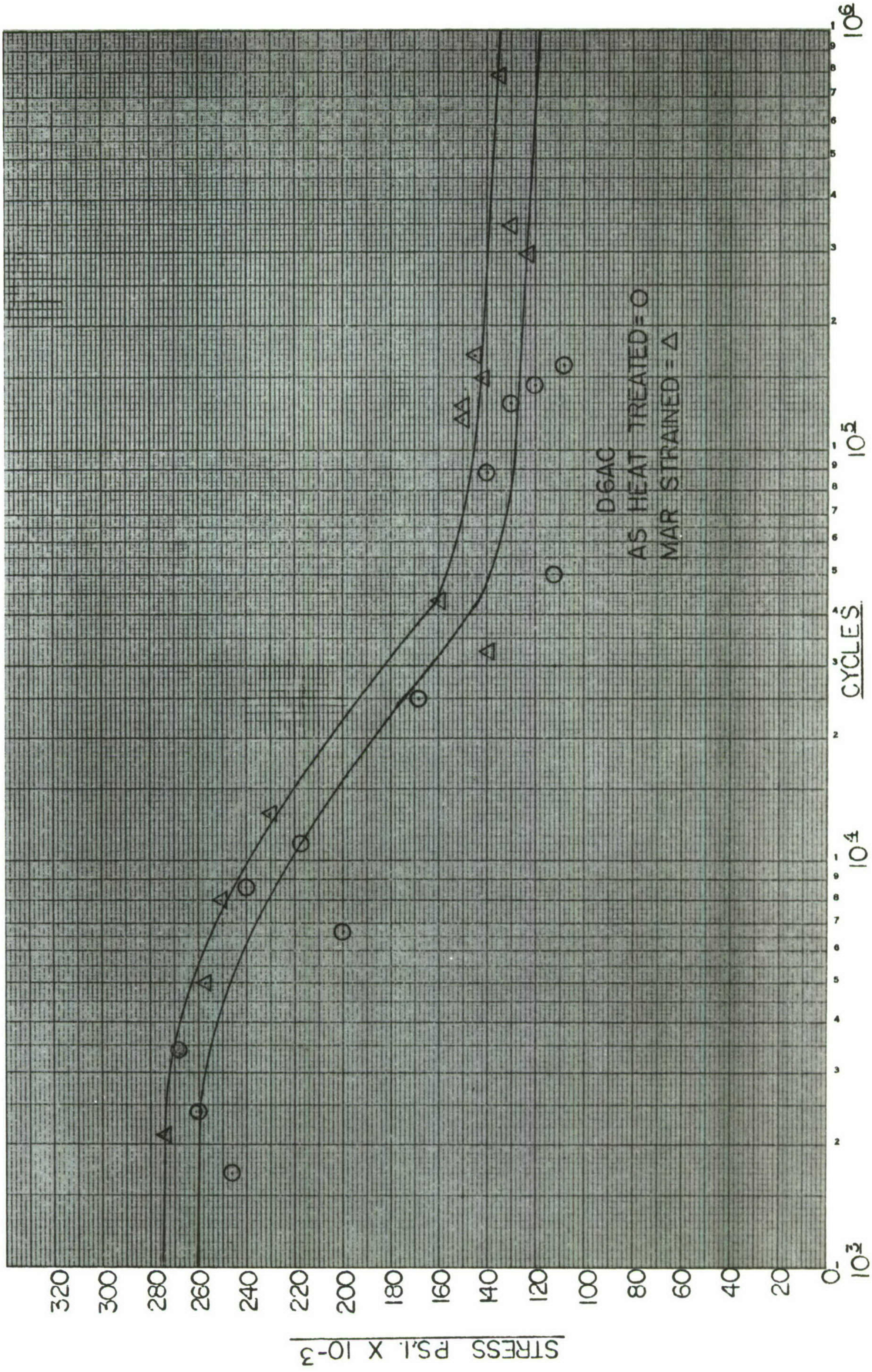



FIGURE 56. AXIAL FATIGUE PROPERTIES D6AC

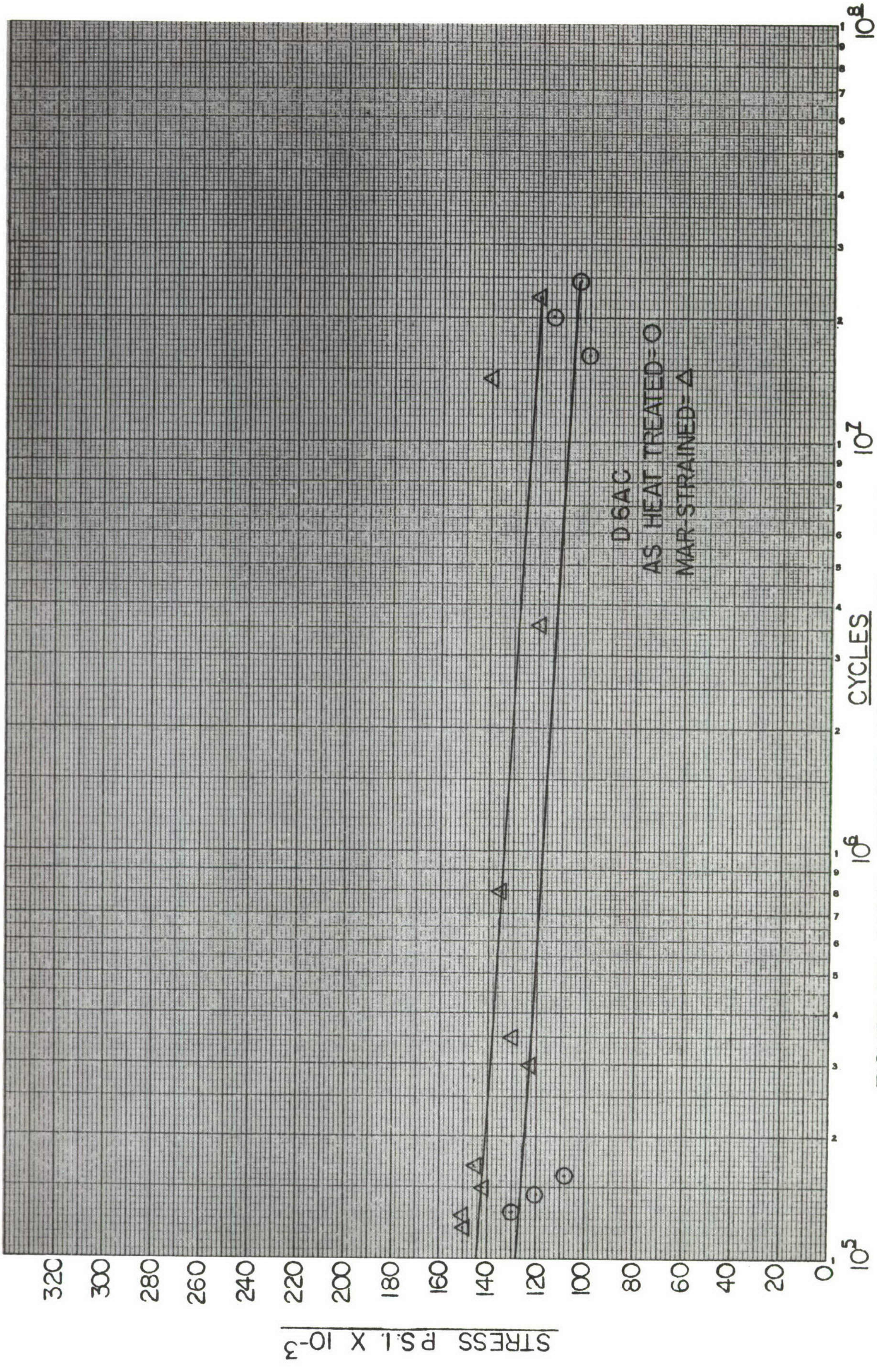



FIGURE 57. AXIAL FATIGUE PROPERTIES D6AC

The drop-off from the knee to the run-out was more gradual for the D6AC. Again an increase in fatigue strength was achieved by Mar-Straining.

Three areas were evaluated statistically, 2,000 to 15,000 cycles, 50,000 to 150,000 cycles and the run-out area.

| Area | Heat Treated | Mar-Strained |
|---|---|---|
| 2000 to 15,000 | | |
| Mean observation | 241,200 psi | 252,200 |
| Average cycles | 5,170 | 7,029 |
| Std. deviation | 22,400 psi | 22,900 psi |
| 50,000 to 150,000 | | |
| Mean observation | 125,400 psi | 147,000 psi |
| Average cycles | 103,000 | 140,875 |
| Std. deviation | 11,900 psi | 8,500 psi |
| Run-Out | | |
| Mean stress | 108,850 psi | 131,600 psi |
| Std. deviation | 22,400 psi | 14,800 psi |
| 95% confidence limits | 85,300 and 132,500 psi | 115,200 and 158,000 psi |

The mean stress at run-out for the Mar-Strained condition was 21% higher than the heat treated condition, approximately the same percentage increase as was accomplished with the Modified S-5.

Bending fatigue data was also obtained for D6AC to give a basis for comparison of the tension-tension data obtained with another type of fatigue loading and to give a basis for comparison of the D6AC heat evaluated under this program with other heats which may have been evaluated elsewhere. The data obtained is tabulated in Table 9 and is plotted in Figure 58. The data indicate that Mar-Straining does not increase the fatigue properties under alternating stress conditions, as might be expected. This would tend to indicate that the Mar-Straining was at least partially a uni-directional phenomenon (such as residual stress or dislocation pinning in one direction only) associated with plastic straining in one direction. The strengthening effects may only be lost after many cycles of stress reversal. Further testing would be required to understand this anomaly fully.

## TABLE 9

## **ALTERNATING STRESS FATIGUE PROPERTIES OF LADISH D6AC

### As Heat Treated

| Spec. No. | Number of Cycles | Stress |
|---|---|---|
| 1 | 38,000 | 149,500 |
| 5B | 117,000* | 114,100 |
| 3 | 134,000 | 124,500 |
| 2 | 407,000* | 125,000 |
| 6 | 665,000* | 113,800 |
| 4A | 2,554,000 | 114,600 |
| 4B | 6,000,000 | 113,700 |
| 5A | 29,309,000+ | 113,200 |

### Mar-Strained

| Spec. No. | Number of Cycles | Stress |
|---|---|---|
| 1 | 46,000 | 136,000 |
| 2 | 69,000 | 127,000 |
| 3 | 135,000 | 120,700 |
| 4 | 10,215,000+ | 113,200 |
| 5 | 243,000 | 116,800 |
| 6 | 17,052,000+ | 115,100 |

* Failure not in gage section - fretting failure in grip

\+ No failure

** Conducted in a sheet bending fatigue machine

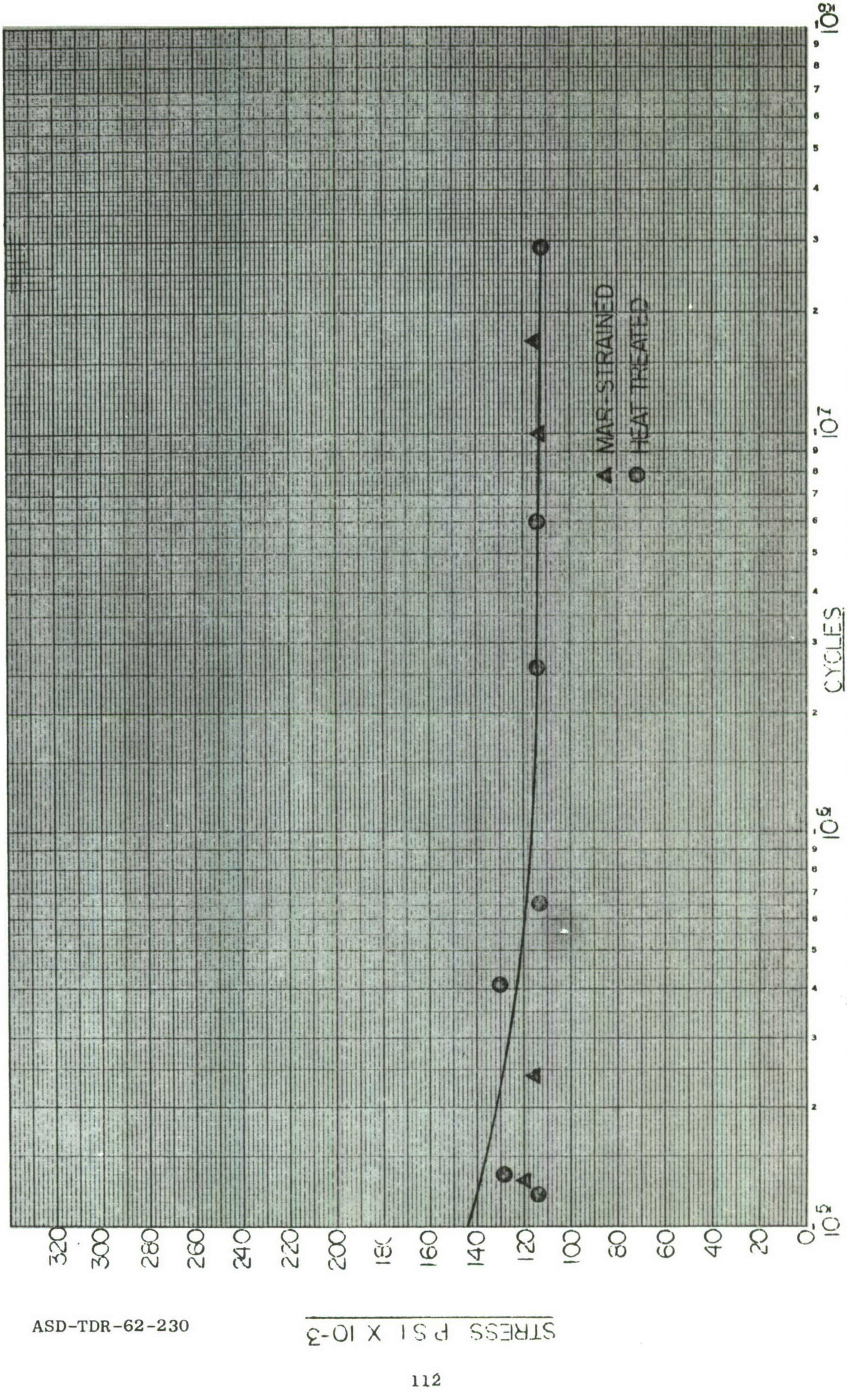



FIGURE 58. ALTERNATING STRESS FATIGUE PROPERTIES OF D6AC

## F.) Biaxial Testing

The biaxial program was planned to confirm the properties obtained in the uniaxial program and to serve to demonstrate the merits of the Mar-Strain process using sub-scale hardware.

### Procedure

Cylinder Preparation:

The ring forgings were machined to .006 to .010 inches (.003 to .005 inches per side) thicker than is shown in Figure 59. This machining operation involved boring the I.D., turning the O.D. contour and final grinding to eliminate tool marks. The cylinders were then heat treated in a Lindberg endothermic gas atmosphere furnace equipped with a Carbotrol unit for control of the dew point on the furnace exit gas. Dew point setting was determined in advance of the heat treatment of the cylinders by heat treatment of several pieces from the same heat of material. Dew points were determined for both the D6AC and Modified S-5 which maintained the decarburization depth between 0 and .002 inches. Heat treat temperatures (austenitizing and tempering) were identical to those used for the tensile evaluation. The cylinders were austenitized in a horizontal position in a cradle which supported the sides of the cylinder around one half of the circumference (Figure 60) to minimize distortion. The cylinder was lifted from the cradle with a specially designed supporting fixture so that quenching could be carried out in a vertical position (Figure 61). Oil was the quenching medium. Tensile specimens were attached to the cylinder during the heat treatment to quality check the cycle if required. A ground specimen (mill scale and decarb removed) was attached to determine the amount of decarburization.

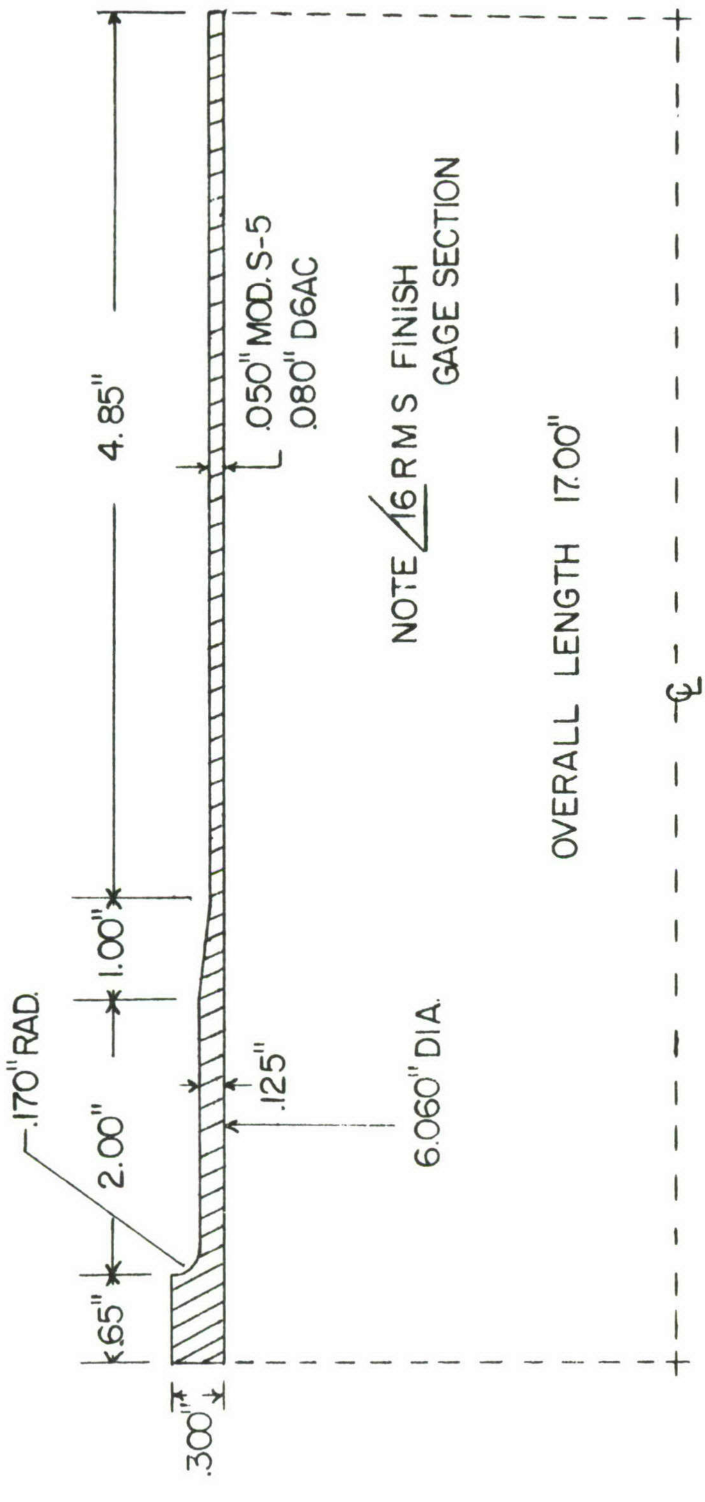



FIGURE 59. BURST CYLINDER

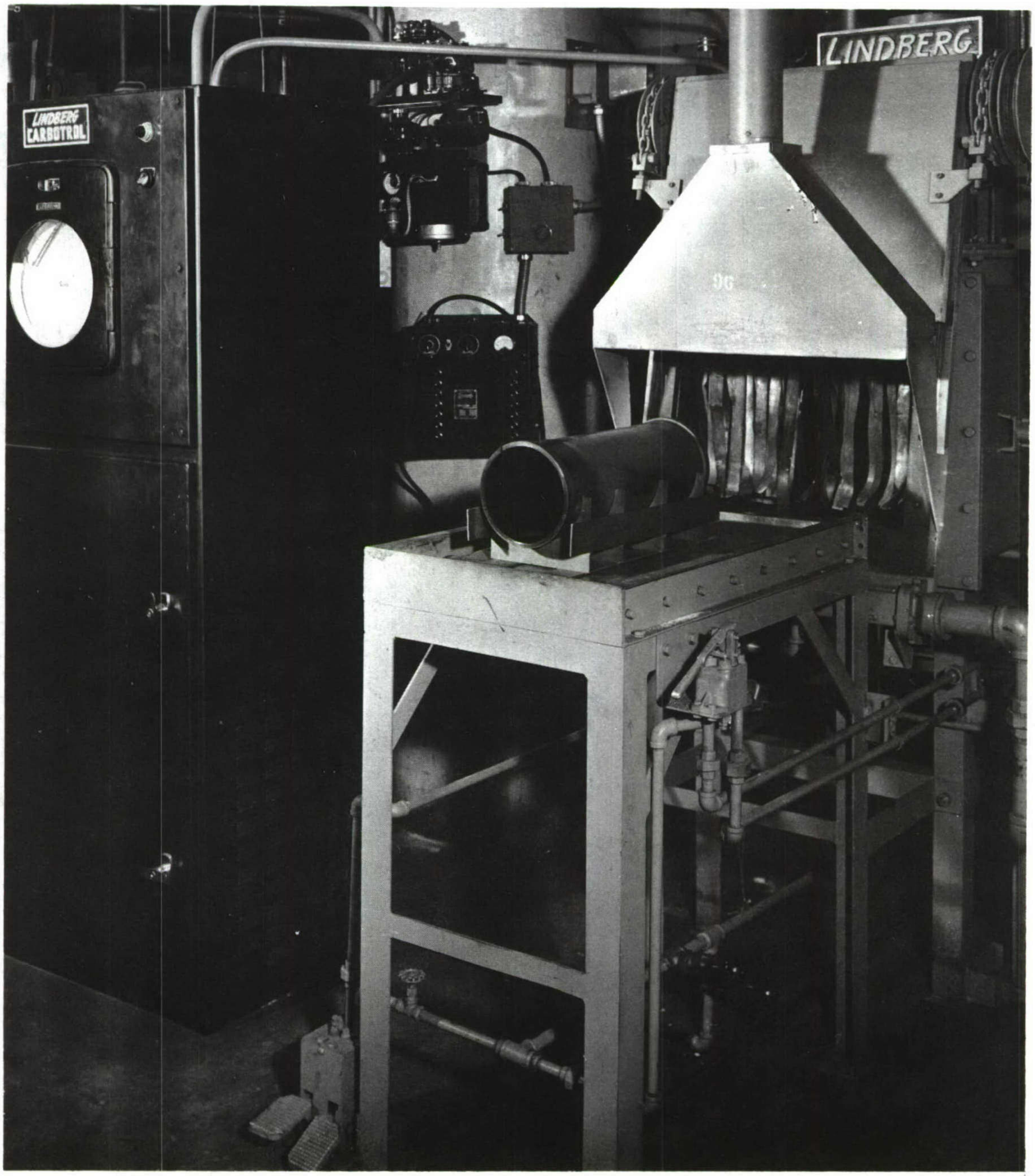



Figure 60. Lindberg Controlled Atmosphere Furnace and 6-Inch Diameter Cylinder in Heat Treat Fixture.

Figure 61. Support Fixture for Vertical Quenching and Oil Quench Tank.

After heat treatment the cylinders were ground inside and out to the desired wall thickness and polished using slow speed grinding. The three stages of cylinder preparation, ring forging, machined and heat treated cylinder,and finally-polished cylinder, are shown in Figure 62.

After final polishing,the cylinders were given a visual inspection for defects. When defects were found, further grinding was employed to remove metal uniformly from the cylinder until the defect was eliminated. The cylinders were then ultrasonically inspected to determine if internal defects were present in the gage section of the cylinder. (The technique was later changed so that the thick portion of the cylinder could be inspected because of a failure resulting from an undiscovered defect in the thick area). Any sizable defects found ultrasonically which were not visible on the surface were located by measurements from a zero reference point and X-rayed to determine the orientation and size of the defect. After this information was obtained, the decision was then made as to whether the cylinder should be tested. Thickness measurements were secured at $90^{o}$ intervals around the cylinder starting at the zero reference. Diameter measurements were taken at $0^{o}$ - 180 and $90^{o}$ - $270^{o}$ locations.

Testing

The testing program was planned to include the preparation of six (6) D6AC and three (3) Modified S-5 cylinders. The D6AC cylinders were to be pre-strained .38% and Modified S-5 .28%. According to the tensile evaluation, these amounts of pre-strain would provide the desired Mar-Strain yield strengths of 275,000 psi and 300,000 psi, respectively. After

Figure 62. Three Stages of Cylinder Preparation Ring Forging, Machined and Heat Treated Cylinder and Finally Polished Cylinder.

Mar-Straining, one of the D6AC cylinders was to be cut up to determine the tensile properties transverse to the direction of plastic strain. At least two of the D6AC cylinders and one of the Modified S-5 cylinders were to be cycled nine times to within 5 to 8% of the desired Mar-Strain yield strength. The remaining acceptable cylinders were to be tested to determine the new Mar-Strain yield and burst strength.

Figure 63 is a display of the equipment used during the pre-straining operation. The cylinder was clamped to the fixture heads. A seal between the heads and the ends of the cylinder was made with a metal O-ring. An internal plug was placed inside the cylinder to reduce the amount of oil required and to reduce the energy to be dissipated when the cylinder was burst. The amount of pre-strain was monitored with strain gages and was controlled with a retaining ring. The retaining ring was constructed of a 304 stainless outer shell and was lined with a plastic material (Kish 312) for contact with the strained cylinder. The plastic ring was expendable, and the inside diameter of the plastic was determined by measurement of the outside diameter of the cylinder and the desired amount of pre-strain. This was determined by assuming that growth of the cylinder under pressure was the desired amount of pre-strain plus .75% elastic strain (e.g., for D6AC, .75%+.38% = 1.13% larger than the cylinder OD equals the plastic inside diameter). The cylinder ready for pre-straining is shown in Figure 64. Two pairs of strain gages (Tatnall foil Type C6 121R2B) were used in monitoring. In each pair, one gage was located to measure the strain encountered in the circumferential direction and one in the axial direction. These rosettes were located 180° from each other

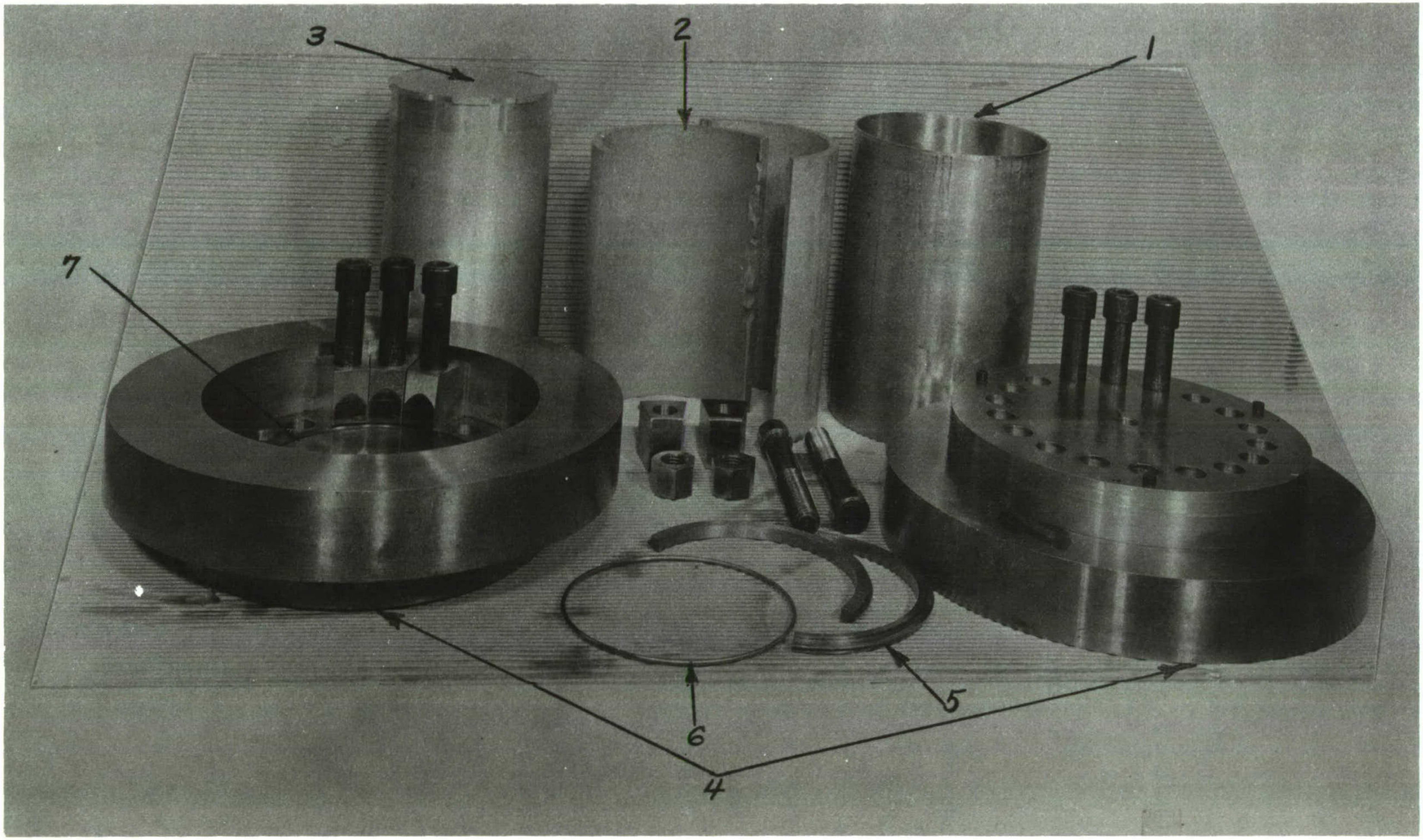



Figure 63. Fixture Equipment Used for Pre-Strain Operation

1. Retaining ring
2. Plastic inner liner
3. Internal plug
4. Fixture heads
5. Snap rings
6. Metal O-rings
7. O-ring groove

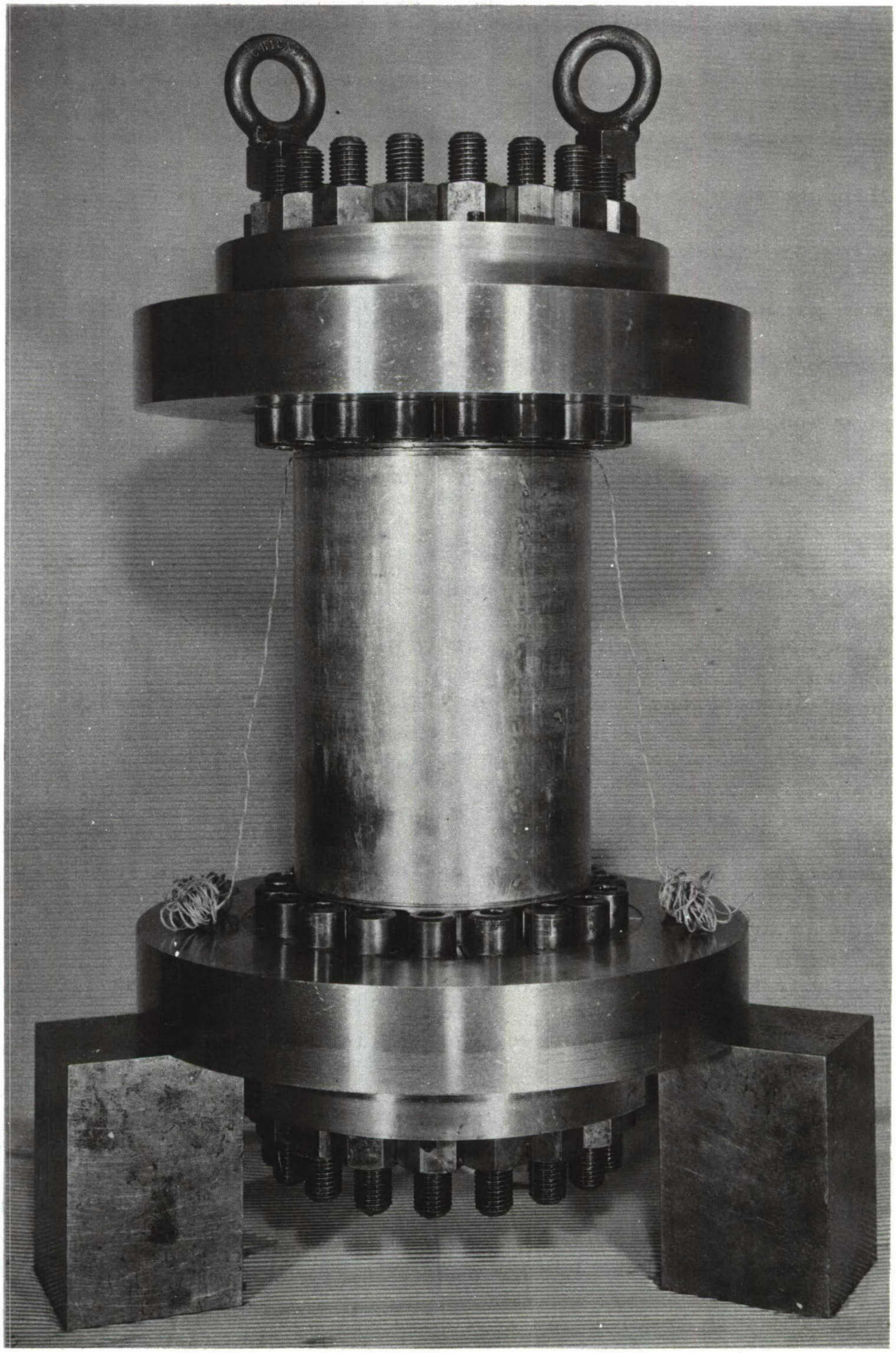


Figure 64. Fixtured Cylinder Ready for Pre-Straining.

and wherever possible were located on the thickest and thinnest spots measured on the cylinder. Strain vs. pressure was recorded on an L and N XX-Y recorder. Oil pressure was supplied by a 30,000# pneumo-hydraulic pressure amplifier positive displacement pump.

After pre-straining, the cylinders were appropriately aged and re-strain gaged. Four rosettes were used (Tatnallfoil Type HE-121). Two pairs of gages were placed at the areas monitored during the pre-straining and the other two were located at other areas of interest. Figure 65 shows a cylinder ready for final burst testing.

Results -

D6AC - Of the six cylinders prepared for testing, one was rejected because of an internal defect found in the gage section during ultrasonic inspection, two cylinders failed during the pre-straining and three were successfully strained, aged and re-tested. Two of the three re-tested were cycled nine times to within 8% of the desired yield strength. The data obtained on these cylinders are tabulated in Tables 10 and 11.

Cylinder No. 1 - This cylinder varied in wall thickness from .071 to .075 inches. Strain gages were placed 180° apart on areas which were .071 and .075 inches in thickness. The stress calculations are based on these thicknesses and the diameters at the gage locations. During the pre-straining operation, the same .2% yield stress (246 Ksi tensile equivalent) was encountered at both areas, but of course, occurred at different pressure levels. At approximately .38% strain, the thin area came in contact with the plastic retaining ring and the strain gage ceased to function. At 6900 pounds pressure, the test was stopped because

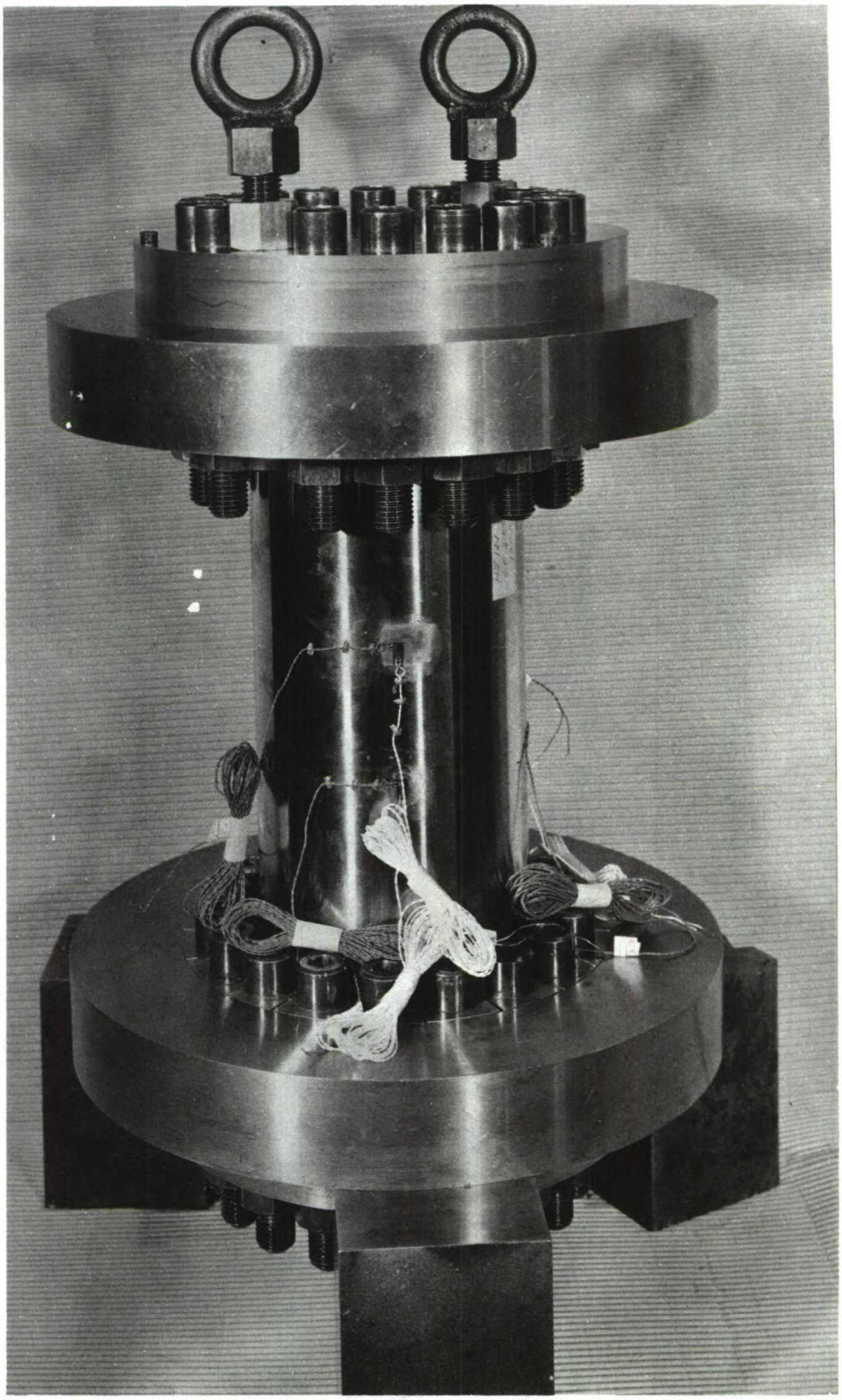


Figure 65. Cylinder Ready for Final Burst Testing.

TABLE 10

PRE-STRAIN DATA-D6AC 6 INCH DIAMETER CYLINDERS

| | | | .2% Yield | | | Final | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Stress-Ksi | | | Stress-Ksi | | |
| Cylinder No. | Wall Thickness (inches) | O.D. (inches) | Pressure Psi | Hoop (1) | Tensile Equivalent (2) | Pressure Psi | Hoop (1) | Tensile Equivalent (2) | Percent Strain |
| 1 | .071 | 6.221 | 6350 | 278 | 246 | - | - | - | .38 |
| | .075 | 6.221 | 6730 | 279 | 247 | 6900 | 286 | 253 | .34 |
| 3 | .077 | 6.223 | 6700 | 271 | 240 | 7200 | 291 | 258 | .38 |
| | .083 | 6.223 | 7200 | 270 | 239 | 7200 | 270 | 239 | .20 |
| 4 | .072 | 6.227 | 6450 | 278 | 246 | 6650 | 287 | 254 | .34 |
| | .078 | 6.221 | - | - | - | 6650 | 265 | 235 | .11 |
| 5 | .070 | 6.207 | 6300 | 279 | 247 | 6550 | 291 | 258 | .35 |
| | .079 | 6.207 | - | - | - | 6550 | 258 | 228 | .08 |
| | .080 | 6.232 | - | - | - | 6550 | 255 | 226 | .04 |
| 6 | .076 | 6.216 | - | - | - | 6600 | 270 | 239 | .06 |
| | .081 | 6.216 | - | - | - | 6600 | 253 | 225 | .03 |

(1) $\text{Hoop-stress} = \dfrac{\text{Pressure X Diameter}}{\text{2 X Wall Thickness}}$

(2) $\text{Tensile Equivalent} = \dfrac{\text{Hoop Stress}}{1.13}$

## TABLE 11

## BURST DATA-D6AC 6 INCH DIAMETER CYLINDERS

| Cylinder Number | Wall Thickness | O.D. | .2% Yield Pressure Psi | .2% Yield Stress-Ksi Hoop(1) | .2% Yield Stress-Ksi Tensile Equiv.(2) | Burst Pressure Psi | Burst Stress-Ksi Hoop(1) | Burst Stress-Ksi Tensile Equiv.(2) | Cyclic Pressure Psi | Cyclic Stress-Ksi Hoop(1) | Cyclic Stress-Ksi Tensile Equiv(2) | No. of Cycles |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | .071 | 6.221 | - | - | - | 7450 | 326 | 288 | 6700 | 293 | 259 | 9 |
| | .075* | 6.221 | - | - | - | 7450 | 313 | 277 | 6700 | 277 | 245 | 9 |
| 3 | Failed during pre-strain | | | | | | | | | | | |
| 4 | .072* | 6.227 | - | - | - | 7200 | 313 | 277 | 6650 | 287 | 254 | 9 |
| | .078 | 6.221 | 7100 | 283 | 251 | 7200 | 287 | 254 | 6650 | 264 | 234 | 9 |
| 5 | .070 | 6.207 | - | - | - | 6550 | 291 | 258 | - | - | - | - |
| | .079* | 6.207 | - | - | - | 6550 | 258 | 228 | - | - | - | - |
| | .080 | 6.232 | - | - | - | 6550 | 255 | 226 | - | - | - | - |
| 6 | Failed during pre-strain | | | | | | | | | | | |

(1) $\text{Hoop-stress} = \dfrac{\text{Pressure X Diameter}}{\text{2 X Wall Thickness}}$

(2) $\text{Tensile Equivalent} = \dfrac{\text{Hoop Stress}}{1.13}$

the gage on the thick area appeared to have reached the .38% offset yield desired. After the cylinder had reached zero pressure, it was found that the actual plastic strain as recorded by the gage was only .34%, but since the cylinder had demonstrated a higher .2% yield strength than the 240 Ksi anticipated, it was predicted that the required 275 Ksi Mar-Strain yield would be achieved with .34% strain, so the test was terminated.

The cylinder was removed from the fixture, aged and again gaged. To demonstrate the cyclic behavior of a Mar-Strained cylinder, a stress 6% below the new expected yield strength (275 Ksi tensile equivalent) was calculated. The cyclic stress was determined to be 259 Ksi (293 Ksi hoop) and, based on the thinnest section, would be achieved at 6700 pounds pressure. The cylinder was cycled to this pressure nine (9) times and on the tenth cycle was held momentarily at 6700 pounds pressure and then taken to burst. Burst occurred at 7200 pounds pressure at a location which was .075 inches in thickness. Only slight signs of yielding were evident from the gages. Initial fracture occurred remote from any gage, but propagated through one of the gages. The hoop burst strength calculated on the basis of the thickness (.075) and the diameter in the area of failure was 313 Ksi (277 Ksi tensile equivalent). The thin area (.071 inches) saw a hoop stress of 326 Ksi (288 Ksi tensile). This was slightly below the predicted yield strength for .38% pre-strained (246 Ksi original heat treated yield strength) D6AC as taken from the yield strength prediction curve (Figure 44B). The fracture was full shear in nature. The burst cylinder is shown in Figure 66 with an arrow denoting the initial fracture origin.

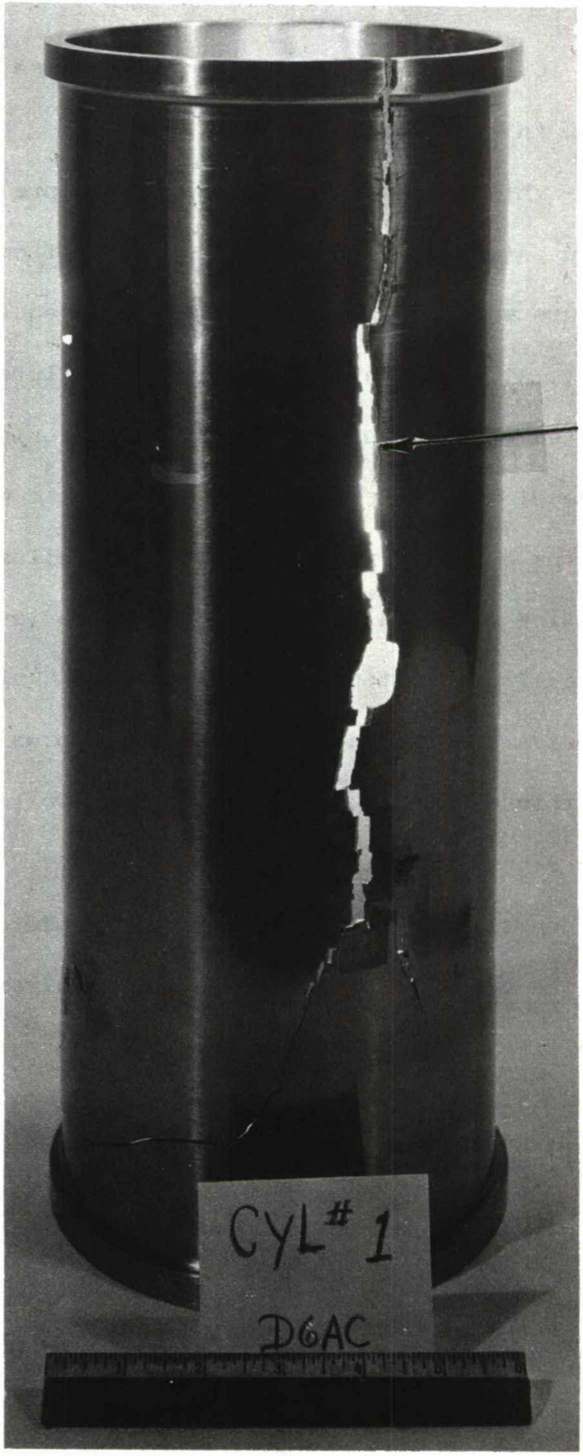



**Figure 66. D6AC Cylinder No. 1 After Burst. Arrow Points to Failure Origin.**

Cylinder No. 2 - Cylinder No. 2 was rejected because of a large ultrasonic indication in the gage section.

Cylinder No. 3 - This cylinder failed during the pre-straining operation at a pressure of 7200 psi. The wall thickness varied from .077 to .083 inches. The stresses at failure were 258 and 239 Ksi, tensile equivalent (291 and 270 Ksi) respectively, at these wall thicknesses. Failure occurred in the gage section at an area which displayed a small ultrasonic indication during inspection. The size of the indication was such that it was believed to be possible to achieve the required pre-strain without failure. (This knowledge was based on information obtained from burst and bulge testing of 300M alloy which displayed better fracture toughness than the D6AC alloy). At the thin wall area, .38% pre-strain was obtained while only approximately .2% strain was achieved in the thicker areas. The fracture was full shear. The burst cylinder is shown in Figure 67.

Tensile specimens were cut from this cylinder to determine the effect of Mar-Straining on the properties transverse to the plastic strain. The specimens were taken from the axial direction and the results obtained were as follows:

.2% yield - 246,500 psi
Ultimate - 275,000 psi
Elongation - 5.5%

Mar-Straining in the hoop direction did not affect the properties in the transverse (axial) direction. The heat treated yield strength of 246,000 psi of the heat treated cylinder was verified, indicating no increase in yield was obtained in the axial direction. This data has important significance when applying the Mar-Strain process for strength increases. In order to obtain a strength increase, the part must be strained in the direction where yield strength increases are required.

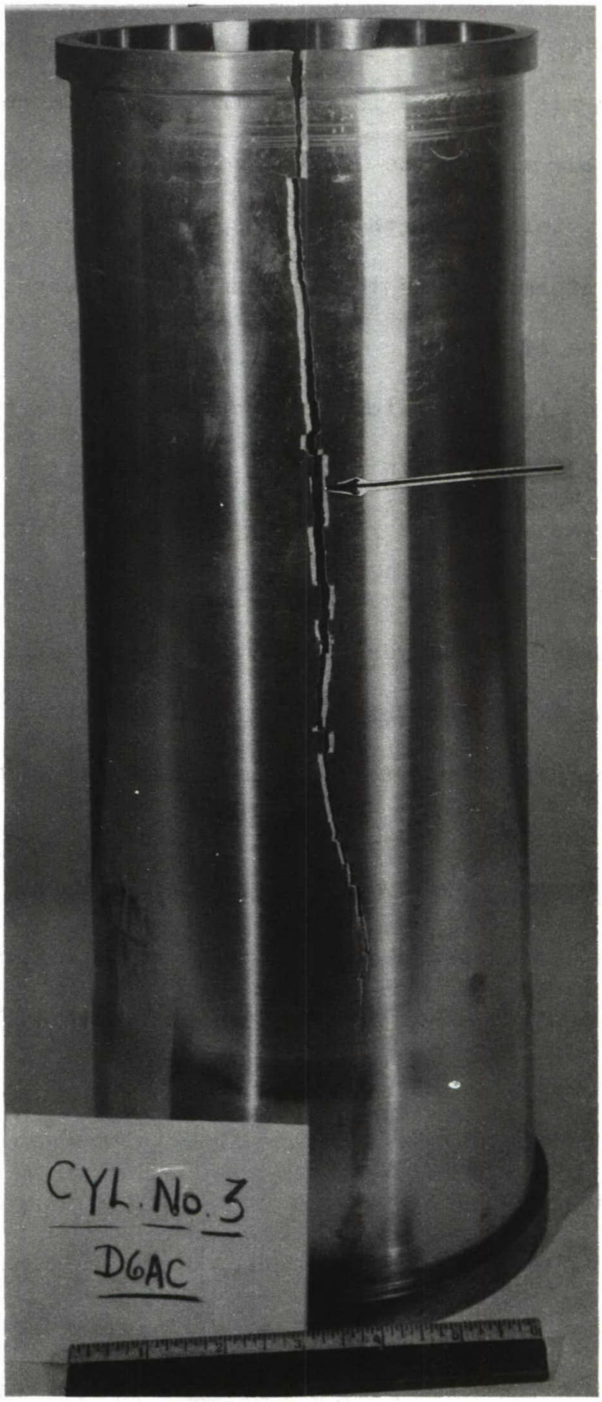



Figure 67. D6AC Cylinder No. 3 After Burst. Arrow Points to Fracture Origin.

Neg. No. C1112209

Cylinder No. 4 - Cylinder No. 4 was not equally pre-strained. .34% strain was obtained in the thin area while only .11% was achieved in the thicker area. The .34% was felt to be adequate for achieving the desired yield strength. Although the thick area was not fully pre-strained, information as to its behavior in subsequent testing was thought to be of value.

Based on the dimensions in the thin area, a stress 8% below the desired 275,000 psi yield strength was chosen for cyclic testing. The cylinder after aging and re-gaging was cycled to this stress nine times and then taken to burst. Yielding (.2%) occurred in the thick portion at 7100 pounds, 100 pounds before the cylinder burst in the thin area. Stress at burst in the thin area was 313 Ksi hoop (277 tensile equivalent), an excellent burst strength for a 275 - 280 Ksi ultimate strength material. The thick area was pre-strained to a tensile equivalent stress of 234 Ksi. The new yield strength was 251 Ksi. The ratio between these stresses was 1.08 which is in fair agreement with the aging response demonstrated in the tensile evaluation (Figure 44A). The burst cylinder is shown in Figure 68.

Cylinder No. 5 - Cylinder No. 5 developed an extremely out of round condition during heat treatment and when subsequently ground for clean-up could not be made uniform in diameter. This non-uniform diameter made it impossible to use the plastic retaining ring for pre-straining. Strain was therefore monitored using three sets of gages instead of the usual two. The thin area was strained to nearly the desired .38% (.35%), whereas the thicker areas were strained only .08% and .04%. After aging and re-gaging, the cylinder was pressurized to burst. The cylinder failed at the same pressure which was incurred during the pre-straining.


Figure 68. D6AC Cylinder No. 4 After Burst. Arrow Points to Fracture Origin.

Evidence of slow crack growth was visible at the fracture origin. The burst cylinder is shown in Figure 69.

Cylinder No. 6 - This cylinder failed during pre-straining at a large defect which was located in the thick portion of the cylinder and out of the gage section. This area had not been ultrasonically inspected. A large length of slow crack was visible as well as evidence of the presence of forging defect. After the failure, all cylinders were inspected along their entire length. The burst cylinder is shown in Figure 70.

Modified S-5 - Of the three cylinders prepared for testing, all were successfully pre-strained, aged and re-tested to burst. One of the cylinders was cycled nine times to within 8% of the desired 300,000 Mar-Strained .2% yield strength. All of the cylinders were pre-strained without the retaining ring because difficulties were incurred in curing the plastic material (Kish 312) sufficiently for proper machining to the desired inside diameter. Detailed test data is listed in Tables 12 and 13.

Cylinder No. 1 - This cylinder varied in wall thickness from .055 to .058 inches. Gages were placed at these thicknesses and the cylinder pre-strained. The thin area was pre-strained .26% and the thicker area .10%. A hoop .2% yield strength of 320 Ksi (283 tensile) was obtained in the thin area. After aging and re-gaging, the cylinder was re-tested to burst. A .2% yield was not encountered at any of the gages. Hoop stress at burst in the thin area near the fracture was 354 Ksi (313 tensile). In the thicker area, the hoop stress was 337 Ksi. Burst performance was excellent; the stress encountered was equal to the tensile strength of the alloy. Although yielding was not measured by strain

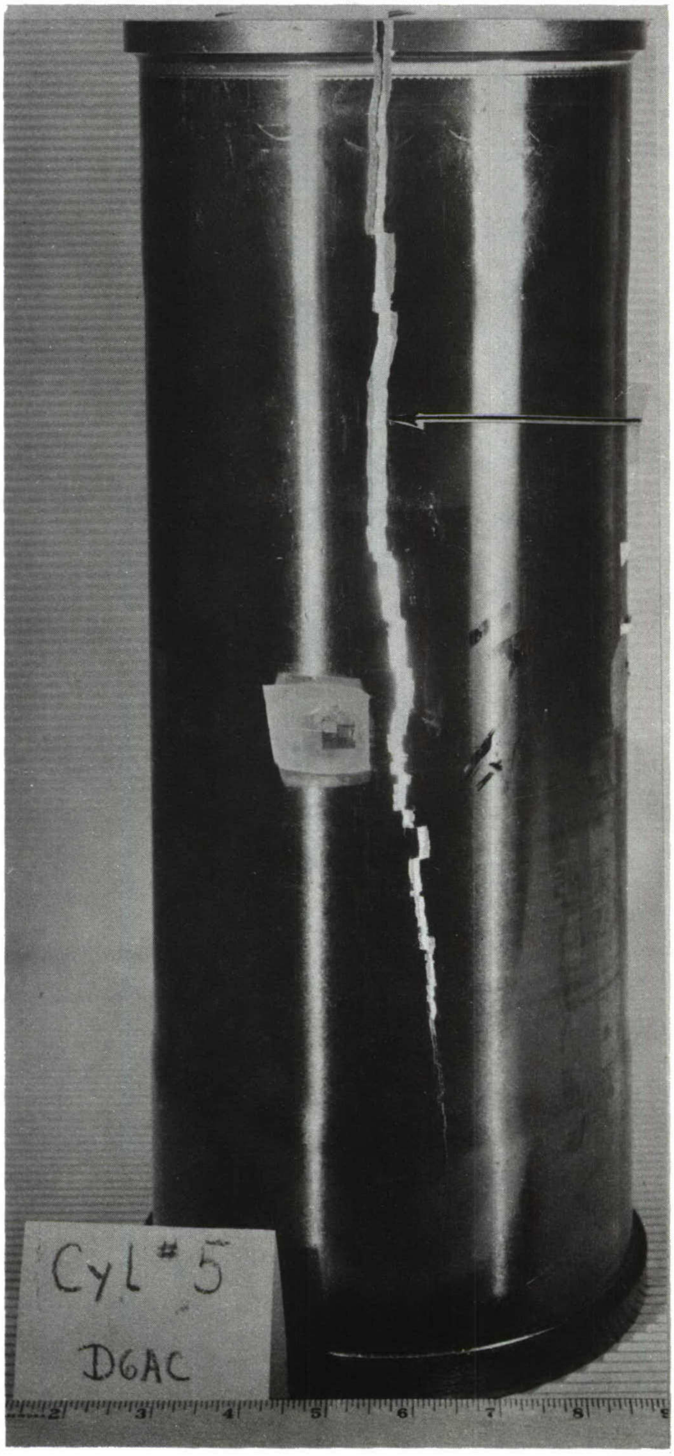



Figure 69. D6AC Cylinder No. 5 After Burst. Arrow Points to Fracture Origin.

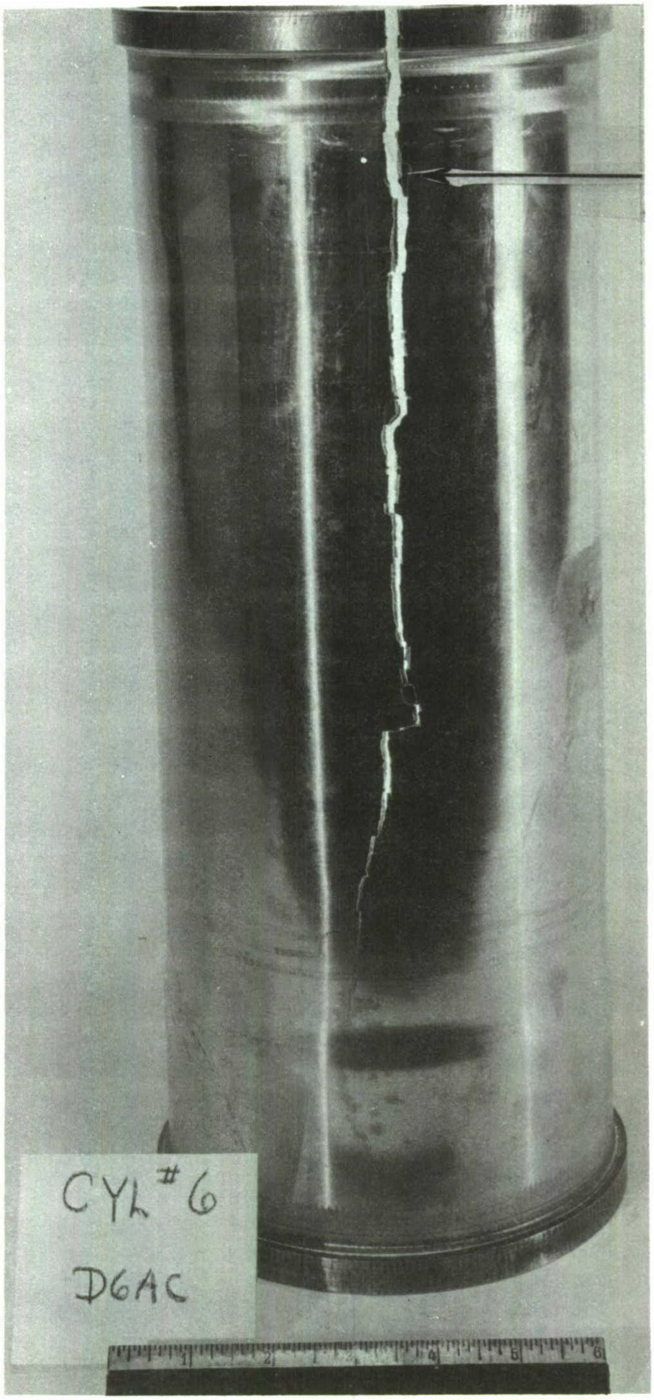



Figure 70. D6AC Cylinder No. 6 After Burst. Arrow Points to Fracture Origin.

## TABLE 12

### PRE-STRAIN DATA-MOD. S-5 6 INCH DIAMETER CYLINDERS

| | | | .2% Yield | | | Final | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Stress-Ksi | | | Stress-Ksi | | |
| Cylinder Number | Wall Thickness | O.D. | Pressure Psi | Hoop (1) | Tensile Equiv. (2) | Pressure Psi | Hoop (1) | Tensile Equiv. (2) | Percent Strain |
| 1 | .058 | 6.207 | | | | 5800 | 310 | 275 | .10 |
| | .055 | 6.183 | 5700 | 320 | 283 | 5800 | 326 | 288 | .26 |
| 2 | .048 | 6.171 | 5000 | 322 | 285 | 5100 | 328 | 290 | .30 |
| | .0545 | 6.172 | | | | 5100 | 289 | 256 | .04 |
| 3 | .0505 | 6.188 | 5200 | 319 | 282 | 5250 | 322 | 285 | .30 |
| | .054 | 6.188 | | | | 5250 | 300 | 266 | .07 |

(1) $\text{Hoop-stress} = \dfrac{\text{Pressure X Diameter}}{\text{2 X Wall Thickness}}$

(2) $\text{Tensile Equivalent} - \dfrac{\text{Hoop Stress}}{1.13}$

## TABLE 13

### BURST DATA-MOD. S-5 6 INCH DIAMETER CYLINDERS

| Cylinder Number | Wall Thickness | O.D. | .2% Yield Pressure Psi | .2% Yield Stress-Ksi Hoop (1) | .2% Yield Stress-Ksi Tensile Equiv. (2) | Burst Pressure Psi | Burst Stress-Ksi Hoop (1) | Burst Stress-Ksi Tensile Equiv. (2) | Cyclic Pressure Psi | Cyclic Stress-Ksi Hoop (1) | Cyclic Stress-Ksi Tensile Equiv (2) | No. of Cycles |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | .058 | 6.207 | - | - | - | 6300 | 337 | 299 | - | - | - | - |
| | .055* | 6.183 | - | - | - | 6300 | 354 | 313 | - | - | - | - |
| 2 | .048 | 6.171 | - | - | - | 5600 | 360 | 318 | - | - | - | - |
| | .0545 | 6.172 | - | - | - | 5600 | 317 | 271 | | | | |
| 3 | .0505 | 6.188 | 5820 | 356 | 315 | 5910 | 362 | 320 | 5100 | 312 | 276 | 9 |
| | .054 | 6.188 | | | | 5910 | 339 | 300 | 5100 | 292 | 259 | 9 |

(1) $\text{Hoop-stress} = \dfrac{\text{Pressure X Diameter}}{\text{2 X Wall Thickness}}$

(2) $\text{Tensile Equivalent} = \dfrac{\text{Hoop Stress}}{1.13}$

gages, pronounced bulging was visible along the fracture area. The fracture was full shear (See Figure 71).

Cylinder No. 2 - The wall thickness of this cylinder varied from .048 to .0545 in thickness. Since the retaining ring was not used, nearly all the yielding occurred in the thin section. A pre-strain of .30% was achieved in this area; whereas, only .10% was encountered in the thicker portion. A .2% yield stress of 322 Ksi hoop (285 Ksi tensile) was achieved in the thin area (in close agreement with cylinder no. 1). On re-testing after aging, a burst hoop stress of 360 Ksi was achieved in the thin area. This stress is equivalent to 318 Ksi in a tensile specimen and is within the spread of values experienced in tensile testing of Modified S-5 specimens. Only slight yielding was recorded at the gage locations, but the fracture area was completely ductile in nature (See Figure 71).

Cylinder No. 3 - The wall thickness was between .0505 and .054. A yield strength of 319 Ksi hoop (282 tensile) was displayed by the thin area. In the thin area, .30% strain was achieved, while .07% was achieved in the heavier area. After aging and re-gaging, the cylinder was cycled nine times at a hoop stress of 312 Ksi (thin area) which was equivalent to 276 Ksi tensile stress, a value 8% below the desired 300,000 psi yield strength. The cylinder was then taken to burst on the tenth cycle. A .2% yield value of 356 Ksi hoop (315 Ksi tensile) was achieved in the thin area. The ratio of the pre-strain stress to the new Mar-Strained .2% yield strength was 1.105, which is in agreement with the aging response values achieved in the tensile evaluation. Yielding approaching .10% was recorded at two other locations. Fracture of the cylinder occurred in an area of the same thickness as the gage which displayed yielding (Figure 71).

Cylinder No. 1 Cylinder No. 2 Cylinder No. 3

Figure 71. Modified S-5 Cylinders 1, 2, and 3 After Final Burst - Arrows Point to Fracture Origin.

A burst hoop stress of 362 Ksi (320 tensile) was achieved in the thin area.

The stresses achieved with the Modified S-5 alloy are the highest values of burst and yield that have been reported to date for a homogeneous material applied to a pressure vessel.

## G.) Conclusions

The investigation of the engineering properties of the two low alloy steels, Ladish D6AC and Modified S-5, has presented several significant conclusions.

1. Although the aging response and strain hardening characteristics affect the magnitude of the strength increase available from Mar-Straining, the major amount of spread in Mar-Strain properties was caused by the inherent spread in the heat treated properties of the alloy.
2. Data obtained by investigating the effect of amount of pre-strain on the tensile properties can be used to establish a yield strength prediction curve which is valuable for predicting the Mar-Strain yield strength if the amount of pre-strain and the heat treated yield strength are known.
3. The notch toughness of the alloys, as measured by notch strength and $K_c$ values, was decreased by Mar-Straining. The maximum decrease in toughness was brought about by as little as .2% pre-strain. This decrease in notch toughness is believed to have been caused by the decrease in strain hardening of the alloy by Mar-Straining. This decrease in toughness is only significant to large defects introduced into the Mar-Strained alloy after

straining and aging. Data in the literature indicate that the Mar-Strain process would be expected to be beneficial in increasing the notch toughness as related to sub-critical defects present in the alloy before straining and aging by the addition of compressive stresses and/or notch blunting during the pre-straining process.

4. Under tension-tension loading, Mar-Straining produced approximately a 20% increase in fatigue run-out stress over the as heat treated condition for both alloys.

5. Mar-Straining was successfully applied to sub-scale pressure vessel hardware. Six cylinders were successfully pre-strained and aged - 3 D6AC cylinders and 3 Modified S-5. Burst strengths of five of the cylinders, on a tensile equivalent basis $\frac{(PD)}{(2T)}/1.13$, were equal to the ultimate strength of the Mar-Strained alloy. One Mar-Strained D6AC cylinder failed at the pressure and stress encountered in pre-straining, however this occurred at a defect.

6. Biaxial yield strengths recorded were equal to the values predicted from the yield strength prediction curve developed by tensile testing.

7. The 350,000 psi hoop burst strengths obtained on the Modified S-5 alloy were higher than any which have been previously reported for a homogeneous material applied to pressure vessels.

8. Three cylinders were successfully cycled to within 8% of the desired Mar-Strain yield strength without failure.

9. The D6AC alloy was more difficult to Mar-Strain because of its lower notch toughness and the presence of internal defects in the prepared cylinders.

10. Although not used for every cylinder, a retaining ring can be used successfully to control the amount of pre-strain in a structure of non-uniform thickness,such as a solid fuel rocket case.

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## VIII. APPENDIX

### Staircase Analysis (Sample Calculation)

**Mean Fatigue Limit**

$$m = S_o + d\left(\frac{A}{N} \pm \frac{1}{2}\right)$$

+ 1/2 for analysis using run-outs
- 1/2 for analysis using failures

where $S_o$ = lowest stress of run-out (or some assumed baseline)
d = step interval (KSI)
A = $\Sigma$ i Ni
N = $\Sigma$ Ni
i = step value above $S_o$ (i.e., number of steps)
Ni = number of failures (or run-outs, depending on type of analysis) at the "i" th stress level.
i Ni = product of i and Ni at each level
$i^2$ Ni = product of $i^2$ and Ni at each level

**Standard Deviation**

$$\sigma = 1.62\, d \left[\frac{NB-A^2}{N^2} + 0.029\right] \qquad \text{where } B = \Sigma\, i^2\, Ni$$

**Estimate of 95% Confidence Limits**

Confid. Limits = $m \pm 1.96 S_m$
where $S_m$ = standard error of the mean

$$SM = \frac{G\sigma}{\sqrt{N}}$$

when G = 0.95 for $\frac{d}{\sigma}$ = 0.5
G = 1.00 for $\frac{d}{\sigma}$ = 1.0
G = 1.05 for $\frac{d}{\sigma}$ = 1.5
G = 1.10 for $\frac{d}{\sigma}$ = 2.0
and other values interpolated between these

## Endurance Limit - Modified S-5 (as heat treated)

Staircase analysis using run-outs and stress increments of 2500 psi.

| Spec. No. | Cycles X 1000 | Stress KSI | i | Ni | iNi | $i^2Ni$ |
|---|---|---|---|---|---|---|
| | | 107.5 | 0 | 0 | 0 | 0 |
| 12 | 13,521 | 110 | | | | |
| 14 | 10,018 | 110 | 1 | 2 | 2 | 2 |
| 7 | 2,242 | 115 | 3 | 1 | 3 | 9 |
| 9 | 1,010 | 117.5 | 4 | 1 | 4 | 16 |
| | | | | 4 =N | 9 =A | 27 =B |

**Mean**

$$m = S_o + d\left(\frac{A}{N} + 0.5\right)$$

$$= 107.5 + 2.5\left(\frac{9}{4} + 0.5\right) = 107.5 + 2.5\ (2.75) = 107.5 + 6.88$$

$$= 114.38 \text{ KSI}$$

**Std. Dev.**

$$\sigma = 1.62\ d\left[\frac{NB-A^2}{N^2} + 0.029\right] = 1.62\ (2.5)\left[\frac{108-81}{16} + 0.029\right]$$

$$\sigma = 1.62\ (2.5)\ (1.717) = 6.954$$
$$\sigma = 7.0 \text{ KSI}$$

**95% Confidence Limits**

$$= m \pm 1.96\ S_m$$

$$= m \pm 1.96\ \frac{G\sigma}{\sqrt{N}}$$

$$\frac{d}{\sigma} = \frac{2.5}{7.0} = 0.36$$

$$= 114.4 \pm \frac{1.96\ (0.94)\ 7.0}{2}$$

$$= 114.4 + 6.45$$
$$= 120.9\ \&\ 107.9 \text{ KSI}$$

Fatigue Strength - Modified S-5 (as heat treated) 2000 to 7000 Cycles

Choosing those specimens which failed from 2000 to 7000 cycles.

| Spec. No. | Cycles | X Stress KSI | X2 |
|---|---|---|---|
| 10 | 3000 | 273 | 74,529 |
| 6 | 4500 | 240 | 57,600 |
| 1 | 3300 | 270 | 72,900 |
| 25 | 4500 | 262 | 68,644 |
| 31 | 6500 | 248 | 61,504 |
| 32 | 3200 | 285 | 81,225 |
| | 25,000 | 1578 | 416,402 |

$$\bar{X} = \frac{\Sigma X}{N}$$

$$\sigma = \sqrt{\frac{\Sigma X^2}{N} - \bar{X}^2}$$

X = observation

$\bar{X}$ = mean observation

$$\bar{X} = \frac{\Sigma X}{N} = \frac{1578}{6} = 273 \text{ ksi}$$

Ave. Cycles $= \frac{25000}{6} = 4200$ cycles

$$\sigma = \sqrt{\frac{\Sigma X^2}{N} - \bar{X}^2} = \sqrt{\frac{416000}{6} - 69169} = \sqrt{69400 - 69169} = \sqrt{231} = 15 \text{ ksi}$$